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SUGARCANE

AND SUGARCANE PRODUCTS

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A Bibliography

of

Research

At The

Southern Utilization

Research and Development Division

Of The

Agricultural Research Service

U. S. Department of Agriculture

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A BIBLIOGRAPHY OF PUBLICATIONS ON THE CHEMISTRY  
AND TECHNOLOGY OF SUGARCANE AND SUGARCANE PRODUCTS

Compiled and Edited  
by  
Marie A. Jones

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SOUTHERN UTILIZATION RESEARCH AND  
DEVELOPMENT DIVISION

For purposes of research by the U. S. Department of Agriculture on utilization of agricultural crops, the country has been divided into four regions. Each region is served by a Utilization Research and Development Division of the Agricultural Research Service. The Southern area includes Alabama, Arkansas, Florida, Georgia, Louisiana, Mississippi, North Carolina, Oklahoma, Puerto Rico, South Carolina, Tennessee, and Texas. Headquarters for the Division are located at:

Southern Regional Research Laboratory  
1100 Robert E. Lee Boulevard  
New Orleans, La.

This Laboratory conducts research on utilization of cotton, cottonseed, rice, tung, peanuts and other oilseeds, sugarcane and sugarcane products, and sweetpotatoes, and on fundamental chemistry and process engineering and development applicable to utilization of these crops. Five field stations in the Division are located near sources of supply of the raw materials being studied, including citrus and other subtropical fruits, pine gum and its derivatives, and cucumbers and miscellaneous southern-grown vegetables. These field stations are:

U. S. Sugarcane Products Laboratory, Houma, La.  
Naval Stores Station, Olustee, Fla.  
U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Fla.  
U. S. Food Fermentation Laboratory, Raleigh, N. C.  
U. S. Fruit and Vegetable Products Laboratory, Weslaco, Tex.

For information on any of the lines of research being conducted in the Southern Utilization Research and Development Division, you are invited to write or visit the Southern Regional Research Laboratory, or the field station immediately concerned with the product in which you are interested.

Single copies of available reprints may be obtained without cost by addressing a request to the Southern Utilization Research and Development Division at the above address.

Copies of patents may be purchased from the Patent Office, U. S. Department of Commerce, Washington 25, D. C., for 25 cents per copy.



## IMPROVED PROCESSING METHODS FOR SUGARCANE SOUGHT

Constant improvement in processing efficiency is vital to the economical utilization of sugarcane produced on the American mainland and insular areas of production. The Southern Utilization Research and Development Division therefore channels the major portion of its sugarcane and sugarcane products research program along lines designed to supply processors with information which will enable them to increase their efficiency of operation.

One of the principal current research projects is aimed at the development of new and improved methods of clarification and processing to increase the recovery of raw cane sugar, and to improve its quality. Clarification of sugarcane juice by treating with lime and heating is the method generally employed. This method is both simple and cheap, and has been in use for many years. A great deal has been learned about composition of the juice, and of the chemical nature and concentrations of non-sugar constituents present. With this knowledge, it should be possible to develop new and more effective methods of clarification. One of the most promising approaches appears to be the addition of a commercial flocculant to improve the precipitation of undesirable constituents from the raw juice and improve filterability. Several flocculants have been tried, in cooperation with the American Sugarcane League, Louisiana State University, and various sugarcane processors. Investigations so far indicate that effectiveness of the flocculants can be improved by the addition of bentonite. Studies on this and other clarification methods are being continued.

While studies of improved processing methods are being carried out in the pilot plant, other researchers at the Southern Division have taken a more fundamental approach to the same basic problem. Determinations of the sugars, and of the percentages of simple constituents for which quantitative analytical methods have been developed, account for approximately 96 to 97% of the total solids of the raw juices which have been analyzed in detail. The remainder is known to consist largely of more complex organic substances, not all of which have been characterized chemically or determined by quantitative analysis. Classification of the non-sugars as alcohol precipitable gums, chloroform extractable lipids and waxes, and protein estimated from the total nitrogen content, has not proved adequate for investigation of their effects upon the economy and efficiency of refining. Significant progress has been made in the identification of complex organic constituents of high molecular weight, providing a better understanding of their importance, and a basis for devising more effective methods for their elimination, either in production of raw sugar or in the refining process.

Closely related to the general problem of improved processing methods is that of filtration of the raw sugars. This difficulty has

been attributed to various causes: mechanical harvesting, which allows larger amounts of these organic non-sugars to enter the filtration stage of the manufacturing process; rainy weather during the harvesting season; deterioration of cane prior to grinding; and various other possible causes have been suggested. It seems apparent, however, that some abnormal factor, or excessive amounts of some of the normal constituents of the cane juice are being carried through to the raw sugar, to interfere with the refining process, thus slowing down the refinery and requiring larger quantities of filter aid.

Several publications reporting results of research on the composition of sugarcane juice and sugarcane products are included in this listing; still others reporting on this and other research on sugarcane will be forthcoming as the work progresses.

In addition to these, there are listed a number of other papers reporting on research accomplishments of interest to members of the sugar industry. These accomplishments are applicable to many different phases of the industry.

One of the most valuable of the recent accomplishments, in terms of immediate financial benefit, perhaps is the study which demonstrated the advantages of prompt grinding of the cane after harvesting.

The ion-exchange process for purification of raw sugar sirups was adapted to the requirements of a candy manufacturer in Puerto Rico for liquid sugar. A large pilot plant has been installed in Puerto Rico for semi-commercial production of the sirup. Researchers from the Southern Division have spent considerable time in Puerto Rico, working in cooperation with the staff of the Agricultural Experiment Station there and representatives of the sugar industry to adapt the process to plant operations.

A method for the production of aconitic acid from sugarcane juice enjoyed some commercial use at one time. A method for production of wax from sugarcane has also been developed.

Most of the work on confectionery products has been conducted in co-operation with the National Confectioners' Association. At the present time this phase of research is concentrated on coatings and fat products for the confectionery industry. A cooperative project for development of a cocoa butter-like fat from domestic oils has made substantial progress, with encouraging results. The industry is also interested in a rapid method for tempering of chocolate, in new fat products for use as slab oils, in some specialized fat products, such as the acetoglycerides, and in a method for direct esterification which should greatly simplify the preparation of fat products with desirable properties for special uses. While this work is outside the area of sugarcane research, it is included in this bibliography because of interest to the confectionery industry.

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CHEMISTRY AND COMPOSITION

2433. THE ISOLATION, SEPARATION, AND IDENTIFICATION OF THE PRINCIPAL PHOSPHOLIPIDES OF SUGAR CANE JUICE  
Friloux, James J.; and Cashen, Norton A.  
J. Agr. Food Chem. 10: 509-511. 1962

This investigation of the phospholipides of sugarcane juice is part of a broad study on nonsugars in cane juice. Because of the emulsifying nature of phospholipides, it is believed that these compounds have a deleterious effect on the processing and clarification of cane juice. By utilizing a combination of solvent fractionation and silicic acid column chromatography, the principal phospholipides were isolated from lyophilized fresh sugar cane juice and separated. They corresponded chromatographically to phosphatidyl ethanolamine and lecithin on silicated glass paper.

2389. SEPARATE DETERMINATION OF GLUCOSE AND FRUCTOSE AND ESTIMATION OF OTHER REDUCING SUBSTANCES IN MOLASSES  
Roberts, E. J.; Jackson, J. T.; Fort, C. A.; and Martin, L. F.  
Intern. Sugar J. 64: 197-201. 1962

Separation of the reducing sugars from sucrose and other reducing substances by partial chromatography on carbon-Celite columns, and determination of glucose by a specific enzymatic method, has confirmed the predominance of fructose in the mixture of the sugars present in commercial molasses from various sources. The proportions of the two sugars were of the same order as those found in samples of Australian molasses by Foster and Marsh employing paper chromatography for complete separation and colorimetric estimation of the individual reducing sugars. The equivalent reducing power of other reducing substances in molasses has been measured by cuprimetric determinations of the sugars and other substances separated by the carbon column procedure. Identification of the other reducing substances will be necessary to account for the excess reducing power, and its increase during enzymatic inversion for determination of sucrose in molasses.

2129. PROGRESS IN DETERMINING ORGANIC NONSUGARS OF SUGARCANE JUICE THAT AFFECT SUGAR REFINING  
Roberts, E. J.; and Martin, L. F.  
Proc. Tech. Sess. Bone Char, 6(1959): 67-99. 1961

The importance of organic nonsugars in affecting refining operations has received increasing attention during recent years. Discussions at the 1957 Technical Session on Bone Char presented evidence that aconitic acid, a major constituent of these nonsugars, decreases the decolorizing capacity of bone char. Concentrations of both nonnitrogenous carboxylic acids and amino acids have been determined during several grinding seasons in raw juices, the material from which raw

sugar is obtained, as part of our investigation of clarification of juices of new sugarcane varieties grown in Louisiana. A summary of results of this work is presented as a guide for further study of this problem in relation to the refining process. The qualitative findings are directly applicable in dealing with the composition of raw sugars even though quantities and proportions of the individual acids will differ from those found in juices, and will not be of the same magnitudes as raw sugars from different sources. The analytical methods that have been applied successfully for investigation of the composition of raw juice solids are described as being adaptable for determination of these impurities in raw sugars and refinery liquors.

1783. SUGAR IMPURITIES. COMPOSITION OF "FLOC" FORMED IN  
ACIDIFIED SIRUPS FROM REFINED GRANULATED CANE SUGARS  
Stansbury, Mack F.; and Hoffpauir, Carroll L.  
J. Agr. Food Chem. 7: 353-58. 1959

Refined granulated cane sugar occasionally contains trace amounts of material which gradually precipitates from acidified sirups. This "floc" is undesirable in bottled beverages. Its major components are starch, lipids (wax), protein, ash constituents, and decolorizing carbon from the refining operation. Carbon and the organic components predominate. Silica is the major ash constituent. Possible test procedures for estimating floc in refined cane sugars were investigated. An improved procedure adapted from a floc test currently used in the beverage industry appears to offer the most promise.

1316. QUANTITATIVE DETERMINATION OF THE AMINO ACIDS OF SUGARCANE  
JUICE  
Roberts, E. J.; and Martin, L. F.  
Sugar 51(1): 32-33. 1956

A method for the quantitative determination of the amino acids in sugarcane juice is described. Three of these acids, threonine, proline, and phenyl alanine, not previously reported in sugarcane juice, have been isolated and identified. Percentages of amino acids determined in samples of juice from two varieties of cane are given.

1245. COLOR FORMATION IN SUGAR SOLUTIONS UNDER SIMULATED  
CANE SUGAR MILL CONDITIONS  
Wolfson, M. L.; Binkley, W. W.; and Schumacher, J. N.  
Ind. Eng. Chem. 47: 1416-17. 1955

These model experiments suggest that at pH 8 one of the main color-producing systems in cane crusher juice is that of D-fructose and D-glucose with asparagine, followed by that of D-fructose and D-glucose with the aconitate ion. The former is a Maillard type of reaction involving interaction of the reducing sugar with the amino group of the amino acid. The second reaction is undoubtedly an alkaline interaction of the reducing sugars, in which the tripotassium

aconitate contributes to the general acid-base environment but does not enter into actual chemical combination with the sugars. Both of these reaction types are under further investigation in this laboratory.

1114. STUDIES OF THE CHEMICAL COMPOSITION OF MATERIALS CAUSING TURBIDITY IN CLARIFIED SUGARCANE JUICES

Fort, C. A.; and Smith, B. A.  
Sugar J. 17(6): 18-21, 32. 1954

Clarified juices of widely varying turbidity were subjected to high-speed centrifuging, and the amount and composition of the sediments obtained were studied. The total sediments ranged from 0.18 to 0.72 percent on dry juice solids, or from 0.45 to 1.80 pounds per ton of cane. Organic substances predominate, averaging 72 percent. About two-thirds of this organic material was analyzed to be lipids, protein, and starch; the other one-third was not identified. The major portion of the ash or mineral portion of the sediment was siliceous. The nonsilica fraction of the minerals contained iron, aluminum, calcium, magnesium, and phosphorus.

1040. IDENTIFICATION AND DETERMINATION OF NONNITROGENOUS ORGANIC ACIDS OF SUGARCANE BY PARTITION CHROMATOGRAPHY

Robert, E. J.; and Martin, L. F.  
Anal. Chem. 26: 815-18. 1954

Application of partition chromatography on silicic acid columns of large capacity has effected the separation of nine nonnitrogenous acids from sugarcane juice. Acids hitherto undetected--mesaconic, fumaric, and succinic acids--have been isolated and identified, and more satisfactory confirmation of the malic, oxalic, citric, and glycollic acids has been obtained. Previously, only aconitic acid, the predominant compound of this class, has been studied extensively. The method of Marvel and Rands employing their small standard columns has also provided a tool for studying effects of the variety of cane, conditions of its growth, maturity, and other factors on composition and quality of juice for recovery of sugar.

919. AMINO ACIDS IN CANE JUICE AND CANE FINAL MOLASSES

Kowkabany, G. N.; Binkley, W. W.; and Wolfrom, M. L.  
Agr. and Food Chem. 1: 84-87. 1953.

Cane blackstrap molasses contains as much as 50 to 60 % simple sugars. Recovery of these sugars in sucrose production is not economical because of the presence of substances formed from reaction of the sugars with amino acids of sugar cane juice. Chromatographic procedures were applied to this juice and its corresponding molasses to identify the responsible amino acids. Two-dimensional, ascending paper chromatography indicates the probable presence of asparagine, aspartic acid, glutamine, glutamic acid, glycine, alanine, valine,

leucine (or isoleucine), serine, tyrosine, and  $\gamma$ -aminobutyric acid in Florida cane juice and (except serine, tyrosine, and glutamine) in its corresponding final molasses.

892. FURTHER NOTES ON STARCH IN LOUISIANA CANES, JUICES,  
AND RAW SUGARS

Balch, R. T.

Sugar J. 15(8): 11-15.

1953

Forty-one preharvest samples of 1949 cane from twelve varieties all contained starch which usually deposited in a restricted area of every sound node of the millable stalk. Twenty-six samples of commercially milled juice representing nine varieties of 1951 cane all contained starch. Starch was found in all weekly samples of raw sugars from seven factories collected through the 1951-52 season; the amount of starch decreased with the advance of the season, apparently reflecting the decrease in raw juice and resulting from freeze damage to the cane supply.

824. NOTE ON THE STARCH CONTENT OF LOUISIANA SUGARCANE  
AND RAW SUGAR

Balch, R. T.; Smith, B. A.; and Martin, L. F.

Sugar J. 15(6): 39-40. 1952

A method which is specific and permits reliable estimates of percentages of starch in properly prepared samples of sugarcane, juices, and sugar, has been used to test four samples of plant cane and one of stubble. Starch contents ranging from 0.0008 to 0.0063 percent on the basis of the whole sample were found for the plant canes and a content of 0.0003 percent was found for the stubble.

361.2 THE SUGARCANE TRASH PROBLEM FROM A CHEMICAL STANDPOINT

Balch, R. T.; and Broeg, C. B.

Sugar 43(12): 31-5. 1948

Trash is a source of considerable amounts of nonsugars; the content of nonsugars, based upon that obtained from clean cane, may be increased by trash as much as 80 percent and it is certain that any increase in nonsugars in juice results in a lowering of recoverable sugar. From a juice clarification standpoint, the extractable impurities from dry leaves are more objectionable than those from other types of trash, since the juices from them are not clarified by lime and heat. Juices from the green leaves and leaf roll clarify well with lime and heat but remain extremely dark-colored. The juices from immature tops vary in their behavior with lime and heat; some clarify well whereas others do not, the difference apparently being associated with variety of cane. These analyses did not reveal the reason for the difficulties which factories experience with slow-settling clarification muds in milling trashy cane during periods of wet weather. The possible effect of such factors as soil in the cane juices and of the deterioration of

trash and of burned cane was not determined. Aconitic acid was shown to be an important constituent of the soluble solids from cane trash. A large proportion of the nonsugars from both trash and clean cane has not been identified.

### 325.6 QUANTITATIVE METHOD FOR ACONITIC ACID AND ACONITATES

Roberts, E. J.; and Ambler, J. A.

Anal. Chem. 19: 118-20. 1947

A method is described for determining aconitic acid and aconitate by decarboxylation in a boiling potassium acetate-acetic acid solution. The determination may be completed in one and one-half hours for dry samples or in two and a half to three hours for solutions. Interfering substances have been studied and are discussed. The decarboxylation is quantitative and the accuracy of the method is dependent on the accuracy of determining carbon dioxide.

### 325.5 THE EFFECT OF pH ON THE STABILITY OF CIS-ACONITIC ACID

IN DILUTE SOLUTION

Ambler, J. A.; and Roberts, Earl J.

J. Org. Chem. 13: 399-402. 1948

Tristrontium cis-aconitate tri- and hexahydrates have been prepared. The hexahydrate precipitates almost immediately from cold solutions, and the presence of large excesses of soluble trans-aconitates has no significant effect on its precipitation. It has been used to demonstrate that, in dilute solutions at pH 7 to 10, cis-aconitates are stable at temperatures up to 90°. Isomerization to trans-aconitates takes place at lower and higher pH values. Only in highly alkaline solutions does increase in temperature cause extensive increase in the amount of cis-aconitic acid or aconitate transformed.

### 325.4 NONINTERFERENCE OF PECTINOUS SUBSTANCES IN ACONITIC ACID

BY ACONITIC ACID METHOD AND OF ACONITIC ACID IN  
URONIC ACID METHOD

Ambler, J. A.; and Roberts, E. J.

Anal. Chem. 20: 880. 1948

Inasmuch as galacturonic acid is decarboxylated in the potassium acetate-acetic acid reagent for aconitic acid, it was expected that the uronic acids would interfere when the method for aconitic acid was applied to sugarhouse materials. However, no interference of this nature has yet been found. Polyuronic acids and derivatives such as pectic acid and pectin are insoluble in the reagent and are not decarboxylated by it. It was desirable to test the method with a uronic acid derivative which is soluble in acetic acid and basically similar in structure to pectic acid. Results of experiments are described.

## 325.3 DETERMINATION OF ACONITIC ACID IN SUGARHOUSE PRODUCTS

Ambler, J. A.; and Roberts, Earl J.

Anal. Chem. 19: 877-78. 1947.

Heretofore the only method for determining aconitic acid in sugarhouse products has been too time-consuming for practical use in the sugarhouse. The decarboxylation method for determining aconitic acid has been successfully applied to sugarhouse and sugar refinery products. The only interfering substance found was sulfur dioxide, which was readily removed from the decarboxylation gases by washing with potassium dichromate solution. Table is given for aconitic acid in sugarhouse products determined by decarboxylation and by extraction.

## 325.2 INVERSION OF SUCROSE AND OTHER PHYSIOLOGICAL CHANGES IN HARVESTED SUGARCANE IN LOUISIANA

Lauritzen, John I.; Balch, R. T.; and Fort, Charles A.  
U. S. Dept. Agr. Tech. Bull. No. 939. 1948

Data is presented that deals with the effects of maturity of cane on inversion and with the effect of temperature, humidity, and loss of moisture on germination (rooting and sprouting), loss of solids, inversion of sucrose, and other physiological changes in hand-harvested and hand-stripped cane of different varieties. The literature is reviewed, storage conditions discussed as well as varieties, sampling, methods of analysis, analysis on cane basis, experimental data, moisture conditions, etc., followed by discussion and conclusions. Seventeen tables illustrate the experiments.

## 922.2 DETERMINATION OF ACONITIC ACID IN MIXTURES WITH CITRIC ACID

Ambler, J. A.; and Roberts, E. J.

Anal. Chem. 19: 879. 1947

The extent of the interference of citric acid in the decarboxylation method for determining aconitic acid is greatest in absolutely anhydrous acetic acid reagent and decreases in the presence of small amounts of water. Boric acid dissolved in the anhydrous potassium acetate-acetic acid reagent prevents the decarboxylation of citric acid but not that of aconitic acid, making possible the determination of aconitic acid in the presence of citric acid.

## 111.2 COMBINED ABSORPTION AND TITRATION TUBE FOR VOLUMETRIC DETERMINATION OF CARBON DIOXIDE

Roberts, Earl J.

Anal. Chem. 19: 616-17. 1947

Although many methods have been described for carbon dioxide determinations, most of them are cumbersome and time-consuming. An apparatus that permits determinations of carbon dioxide to be made accurately and rapidly is described and pictured. It consists of an absorption

tube, a bubbler, and a stirrer. The method of operation is detailed, and some typical results obtained are reported.

325.16 A STUDY OF YEAST GROWTH-PROMOTING SUBSTANCES IN WHITE SUGAR  
 Hall, H. H.; Paine, H. S.; and Fabian, F. W.  
 Food Research 12: 99-110. 1947

A study of the yeast growth-promoting properties of beet and cane sugar has shown that varying amounts of substances are present in different sugars. A substance identified as biotin was found to be present in amounts from 0.025 to 0.445 micromilligram per gram of sugar. Although there is no exact correlation of the amount of growth-promoting substances with other known impurities of the sugar, there is a correlation of averaged yeast inoculum-multiple values with averaged ash values. The growth-promoting substances, like most other nonsugar impurities, are located at or near the surface of sugar crystals and can be effectively diminished by controlled quantities of additional wash water while the massecuites are being centrifuged.

840.1 THE PRODUCTION OF ITACONIC ACID FROM THE CRUDE ACONITATE  
 OBTAINED FROM SUGARCANE MOLASSES  
 Ambler, J. A.; Roberts, E. J.; and Weissborn, F. W., Jr.  
 U. S. Dept. of Agr., Bur. Agr. Ind. Chem., AIC 132.  
 1946. (Processed)

Aconitic acid was first noted in sugarcane products in 1877 but the pyrolytic decomposition of this acid to itaconic acid has had no industrial application because aconitic acid until recently has entered commerce only in small amounts as an expensive dehydration product of citric acid. Recently the experimental production of itaconic acid by fermentation of carbohydrates has been announced. The chemistry of the process is described and laboratory studies on the decarboxylation of aconitic acid and aconitates are given in detail. The preparation of the aconitate from molasses is outlined with results of the laboratory tests. A summary of yields and recoveries of itaconic acid is included. The study shows that the decarboxylation itself is feasible and that the main points for future study concern the isolation and purification of the acid, and possibly the recovery of the citraconic acid formed during the reaction.

229.1 VARIABLE MINERAL COMPOSITION OF BLACKSTRAP MOLASSES  
 Fort, Charles A.  
 Sugar, 41(11): 36-7. 1946

This report covers some work incidental to studies made on the variable composition of sugarcane juices of Louisiana. In 1937 samples of blackstrap molasses were furnished by eight factories manufacturing raw cane sugar by the usual lime clarification process. These represented a fair cross section of production in the Louisiana sugar belt, and the comparative nonsugar compositions reflect the regional differences which were observed in the raw juice analyses. In 1940, a comparison was made of the season's composites of blackstrap from a

Louisiana and a Cuban factory. The analyses made were primarily for the mineral constituents, but such accessory data as was available or determined is included.

325.7 EXTRACTION OF ACONITIC ACID FROM SORGO

Ventre, E. K.; Ambler, J. A.; Henry, H. C.;  
Byall, S.; and Paine, H. S.  
Ind. Eng. Chem. 38: 201-4. 1946.

The recovery of aconitic acid from sorgo juices is increased from 42.9 percent (by usual lime defecation) to 81.7 percent of the available aconitic acid by defecation with lime and calcium chloride. The acid is prepared as tricalcium aconitate (containing 13 to 14 percent of the magnesium salt) in the optimum pH range of 6.5 to 6.7. A maximum of 29.53 percent of the available aconitic acid may exist as the free acid. Juices defecated with lime and calcium chloride gave syrup with slightly greater purity than the usual lime defecation.

325.8 ACONITIC ACID IN SUGARCANE PRODUCTS

Balch, R. T.; Broeg, C. B.; and Ambler, J. A.  
Sugar 40(10): 32-35. 1945.  
Intern. Sugar J. 48: 186-87. 1946.

The juices of Co. 290 cane have the highest content of aconitic acid. Juice from immature cane or the immature portions of the stalk contains three to five times as much aconitic acid as juice from mature cane. Juice from leaf blades and sheaths contains as much as the juice from the tips. There is loss of aconitic acid in the clarification process. It is concentrated in the syrup and eventually in the molasses. The sediment from diluted molasses contains 16 to 35% aconitic acid as the calcium salt. Established yields from 112 tons of green matter per acre are 800 pounds of aconitic acid.

325.9 SOME SALTS OF ACONITIC ACID

Ambler, J. A.; Turer, J.; and Keenan, George L.  
J. Am. Chem. Soc., 67: 1-4. 1945. (No reprints)

The insoluble aconitates which separate from sugarcane and sorgo sirups have been identified as calcium magnesium aconitates having the optical-crystallographic properties of dicalcium magnesium aconitate hexahydrate, although they generally contain less than the theoretical proportion of magnesium. It is suggested that they are solid solutions of tricalcium aconitate hexahydrate with either trimagnesium aconitate or dicalcium magnesium aconitate hexahydrate. The preparation and properties of crystalline tricalcium aconitate hexahydrate, tricalcium aconitate trihydrate, calcium sodium aconitate dihydrate, dicalcium magnesium aconitate hexahydrate, magnesium acid aconitate tetrahydrate, and zinc acid aconitate tetrahydrate are described. Optical-crystallographic properties and indices of refraction are given for the above salts and for tricadmium aconitate hexahydrate and monopotassium aconitate.

325.10 REPORT ON ACONITIC ACID STUDIES AT HOUMA, LOUISIANA, STATION  
Balch, R. T.  
Sugar Bull. 23: 197-98. 1945

Investigations of aconitic acid at the Houma station are reported. The study deals primarily with a survey of the aconitic acid content of cane juices as influenced by certain cultural and varietal factors. Distribution of aconitic acid in sugarcane and in factory products is discussed. The aim of the studies is to discover potential commercial sources of this natural acid.

325.17 INVERSION OF SUCROSE IN THE DIFFERENT PARTS OF THE  
SUGARCANE STALK  
Lauritzen, J. I.; and Balch, R. T.  
J. Agr. Research 61(1): 1-16. 1940

Data is presented relating to inversion of sucrose and to changes in moisture content and Brix in the top-, middle-, and bottom-third lengths of the sugarcane stalk when sectioned into thirds and stored and when stored intact and sectioned after storage. Two experiments on cane stored under controlled conditions of temperature and humidity, and three experiments on cane stored under dry conditions in the shade, and the results are described.

325.18 METHOD FOR DETERMINING THE UNIFORMITY OF QUALITY OF  
WHITE SUGARS  
Ambler, J. A.  
U. S. Dept. Agr., Bur. Agr. Chem. and Eng., ACE-40.  
39 pp. 1940. (Processed)

A paper entitled "Methods of Analyzing White Sugars, Massecuites and Juices" was published in 1934. New determinations have been added and others revised. This second paper describes the procedures in use for determining the quality of sugar, massecuites and sugar plant juices and describes the methods and technique for various determinations.

#### PROCESSING

2439. SUGARCANE MUD FILTRATION  
Guilbeau, W. F.; Coll, E. E.; and Jackson, J. T.  
Sugar Bull. 41: 62-66. 1962.

Experiments were conducted to determine filtration rates under various conditions with the addition of bagacillo, lime, and Separan of the muds from sugarcane juice clarification. The results indicated that the rates were affected by composition of the sugarcane and by foreign matter (soil and trash) introduced with the sugarcane, the type of filter media (cloths) used, and the use of Separan AP-30.

Large variations were observed in volume filtered due to the composition of the sugarcane and foreign matter. The volume filtered was higher with the use of Separan AP-30 and the filtering cycle was increased. The type of cloth used had some effect on filtration of the muds.

2371. MILLING AND PROCESSING QUALITIES OF CANE COMBINED IN SHORT PIECES COMPARED WITH WHOLE-STALK BURNED CANE

Coll, E. E.; Davidson, L.G.; Stewart, C.W.; and Guilbeau, W.F.  
Sugar Bull. 40: 212-19. 1962.

The objective was to determine any differences in milling or processing, resulting from harvesting cane in short pieces for immediate processing, compared with the conventional Louisiana method of harvesting whole stalks, burning, and processing two or three days after cutting. Values for cane yields, harvesting losses, cane and juice qualities, milling and processing characteristics, and estimated sugar yields are tabulated. The inherent problems of both harvesting methods are discussed.

2341. STANDARD PROCESSING EXPERIMENTS ON COMMERCIAL AND UNRELEASED CANES DURING THE 1961 LOUISIANA HARVEST

Coll, E.E.; Guilbeau, W.F.; Fort, C.A.; and Jackson, J.T.  
Sugar Bull. 40: 181-185. 1962.

The primary objective of 1961 lime clarification tests was to relate the processing characteristics of two sugarcane varieties, C.P. 55-30 and C.P. 36-13, to those of the control, C.P. 44-101. C.P. 55-30 is the most promising unreleased variety, and C.P. 36-13 is a commercial cane highly resistant to the mosaic virus. The effect of P<sub>2</sub>O<sub>5</sub> content of mixed juice on processing variables is graphically illustrated for two canes.

2197. SEPARAN AP-30 USED IN PILOT-PLANT EXPERIMENT ON SUGARCANE JUICE CLARIFICATION

Coll, E. E.; Jackson, J. T.; and Guilbeau, W. F.  
Sugar Bull. 39: 298-303. 1961.

The paper reports the results of experimental work done by the U. S. Department of Agriculture in their pilot plant located in the Audubon Sugar Factory of Louisiana State University, during the 1960 crop. The pilot plant is set up with two identical 50 gallon-per-hour single-tray clarifiers with a concentration rate in each. Four hundred gallons of mixed juice were held under refrigeration and warmed to 80° F. as used for each run. Runs were divided into two standard lime clarification tests, with and without Separan. Results are reported in two tables, and are adjusted to one ton of cane processed. By using Separan in this system, the wet mud weight per unit was reduced by 14 percent, with an increase of 1.4 percent of the mixed juice weight recovered directly as clarified juice. Separan had no significant effect on the lime requirement, pH drop across the system, processed juice clarity, or total insoluble solids removed in the mud. It did increase the filterability of clarified juice by 25 percent.

2123. PILOT-PLANT CLARIFICATION EXPERIMENTS WITH NEW CANES  
DURING THE 1960 SEASON  
Guilbeau, W. F.; Coll, E. E.; Fort, C. A.; and Jackson, J.T.  
Sugar Bull. 39: 186-91. 1961.

Pilot-plant experiments on mixed juice clarification by simple liming, employing the standard procedure described in previous reports were conducted at the Audubon Factory during the 1960 campaign with a total of 19 samples of unreleased and new commercial varieties, and of the standard variety CP 44-101 used for comparison.

1960. PROCESSING OF NEW COMMERCIAL AND UNRELEASED SUGARCANES,  
RESULTS OF CLARIFICATION EXPERIMENTS DURING THE 1959  
SEASON  
Guilbeau, W. F.; Coll, E. E.; Friloux, J.J.; and Martin, L.F.  
Sugar Bull. 38: 226-233. 1960.

Results of pilot-plant milling and clarification experiments with samples of cane from the 1959 crop are presented. Unreleased varieties tested were C.P. 51/21 and C.P. 53/1, the latter being processed for the first time to obtain preliminary data and stubble cane of new released varieties C.P. 52/68 and N. Co. 310. Tables and graphs are given to permit comparison of these canes with the standard C.P. 44/101 to evaluate their suitability for processing.

1910. RADIOACTIVE TRACER-LABELED GLUCOSE: MODERN TECHNIQUES IN PILOT  
PLANT EXPERIMENTS  
Martin, L. F.; Guilbeau, W. F.; and Binkley, W. W.  
Sugar y Azucar 55(12): 50-52, 54. 1960. (Previously published  
in Proc. Ann. Mtg. Sugar Ind. Technicians 18 (sec.2)).1959.

For a number of years the Sugarcane Investigations group of the United States Department of Agriculture has been engaged in a comprehensive series of cane juice processing experiments under simulated factory conditions. Juice defecations and concentrations are carried out on a pilot-plant scale. Raw sugar and molasses are obtained from these sirups with other suitable pilot-scale units at the Sugarcane Investigations Laboratory. Cognizant of the far-reaching results derivable from the processing of raw juices containing added radioactive sugars, the above Laboratory began a cooperative project in 1958. Seven millicuries of uniformly labeled carbon-14-D-glucose were added to 300 gallons of mixed raw juices which were processed under simulated mill conditions. The processing of the juice with particular emphasis on the sampling of intermediate products was described. The results from preliminary qualitative radio-assays of these products were presented and certain implications discussed.

1887. PROCESSING OF NEW COMMERCIAL AND UNRELEASED SUGARCANES -  
 RESULTS OF CLARIFICATION EXPERIMENTS DURING THE 1958  
 SEASON

Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.

Sugar Bull. 37: 274-83. 1959

(Condensed version: USDA Clarification Tests -  
 Experiments During 1958 Louisiana Crop.)

Sugar J. 22(12): 23-24. 1959.

Experiments to compare clarification results on juices of unreleased and new sugarcanes were carried out at the small clarification pilot plant operated by the Southern Utilization Research and Development Division in the Audubon Sugar Factory of Louisiana State University for the ninth consecutive season. Tests were conducted on four canes, C.P. 44/101 being used as the standard.

1886. MILLING AND PROCESSING CHARACTERISTICS OF C.P. 51/21,  
 COMPARED WITH COMMERCIAL VARIETIES C.P. 44/101 and

N. Co. 310

Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.

Sugar Bull. 38(6): 70-71. 1959.

Results of milling and processing tests during the 1958 season on an unreleased cane variety, C.P. 51/21, in the Southern Laboratory's pilot plant at the Audubon Sugar Factory, are given. It is compared with the two commercial varieties most widely used at present, C.P. 44/101 and N. Co. 310. Although C.P. 51/21 has low mud volume, clarity of the juice is poorer than in the varieties used as the standard, and its fiber content is considerably higher. Only four experiments were run because of the limited amount of cane of this variety available, and additional tests will be made to evaluate this variety in processing.

1699. PROCESSING OF NEW COMMERCIAL AND UNRELEASED SUGARCANE IN  
 COMPARISON WITH THE STANDARD COMMERCIAL CANE IN PILOT-  
 PLANT CLARIFICATIONS

Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.

Sugar Bull. 37(3): 29-42. 1958

Results of pilot-plant processing evaluation tests during the 1957 grinding season on promising unreleased sugarcane varieties are compared with the standard, widely grown commercial cane. Data obtained in the clarification experiments carried out by personnel of the Southern Utilization Research and Development Division are consolidated in graphs and tables. Test results are given on plant canes of unreleased varieties compared with the standard, and on stubble (second growth) canes of previously released varieties.

1698. FRESHER CANE INCREASES SUGAR RECOVERIES AND PROFITS  
FOR BOTH GROWERS AND PROCESSORS  
Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.  
Sugar Bull. 36(24): 343-46. 1958.

Recapitulated results from operations of Louisiana factories for the 14-year period 1939-1952 showed declining percentages of cane sugar recovered. Experiments made during the 1954 and 1955 seasons on the effect of delay in grinding harvested cane under practical field and factory conditions showed a considerable increase in available sugar when the average delay in grinding cane is reduced to less than 48 hours. Fresher sugarcane delivered to mills more promptly after harvesting contributes to better recoveries, and additional improvements in delivering cleaner cane as soon as possible after harvesting should reverse the downward trend. Tables showing results of experiments are discussed.

1710. ACETYLATION AND OXIMATION (Assay for Certain Oxygen-Bearing Groups in the Dialysed Browning Products from Cane Final Molasses)  
Binkley, W. W.  
Intern. Sugar J. 60: 322-23. 1958.

The acetylation of the cane molasses "browning" polymer indicated the probable presence of six or seven hydroxyl groups per repeating unit. Boron trifluoride-acetic anhydride was found to be a suitable solvent-reagent system for this acylation. Distinct primary and secondary reactions were observed during the polymer with methoxyamine hydrochloride. The consumption of 0.53 millimoles of this reagent by one gram of the polymer would suggest the existence of one carbonyl group in every five repeating units.

1714. EVALUATION OF SUGARCANE VARIETIES 1956 AND 1957 GRINDING SEASONS (A Study to Determine Milling Qualities and Characteristics of Four Different Varieties of Plant and Stubble Cane)  
Stewart, Carl W.  
Eng. Expt. Sta. Bull. 63. Dept. Chem. Eng., Louisiana State Univ. 1958.

Pilot-scale milling tests of different sugarcane varieties during two seasons are summarized. Grinding of 2-ton samples individually in the Audubon mill tandem provides data on sugar extraction and loss of sugar in residual bagasse, on the rate and power consumption in relation to fiber content, quantity of recoverable sugar, and other factors affecting economy and efficiency of milling the different canes. Results obtained in experiments with three unreleased canes, and three canes released most recently for commercial planting are compared with those of comparable tests of samples of the most widely planted commercial cane.

1548. NEW SUGARCANES TO IMPROVE PROCESSING IN LOUISIANA  
Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.  
Sugar J. 20(1): 25-27, 39-40. 1957.

The three sugarcane varieties released most recently to Louisiana growers were evaluated in pilot-plant milling and clarification experiments. In clarification, results obtained in 1956 were similar to those in earlier seasons when variety C.P. 48/103 was superior to the standard, widely grown C. P. 44/101 in ease of clarification, and equal to it in clarity. C.P. 47/193 was more difficult to clarify, but gave juice of better clarity, while N. Co. 310 proved about equal to the standard in clarification.

1546. THE ACTION OF GLYCOL-SPLITTING REAGENTS  
Binkley, W. W.  
Intern. Sugar J. 59(703): 178-80. 1957

Distinct primary and secondary reactions occurred in the oxidation with sodium metaperiodate of the "browning" polymer of cane final molasses. Overoxidation was markedly less when a hydrogenated modification of the polymer was used. One gram of polymer consumed 6.0 millimoles of oxidant with the concomitant production of 0.5 millimoles of formaldehyde and no formic acid. These data favored the (1→6) and (2→6) furanose structures for the hexose residues in the molasses "browning" products.

1477. FACTORS AFFECTING FILTRATION TEST FOR SUGARS  
Balch, R. T.  
Ind. Eng. Chem. 49: 904-06. 1957.

The filtering characteristics of diatomaceous silica are affected by the presence of various salts in aqueous or sugar solutions. This accounts partially for more rapid filtration of solutions of high ash content sugars reported by Kortschak. Filterability increases with concentration and with the valence of the cation of the chlorides added to solutions of refined and raw sugars, at about pH 6. At lower pH values filterability of a number of raw sugars increased, but the same sugars showed increases, decreases, or substantially no change in filterability in solutions that were neutral or adjusted to pH 8. It is important to establish the quantitative effect of salts and pH upon filterabilities of sugar solutions determined with filter aids.

1315. DISTRIBUTION OF STARCH IN (VEGETABLE CARBON) REFINING  
OF CANE SUGAR  
Balch, R. T.; and Friloux, J. J.  
Sugar J. 18(12): 79-80. 1956.

This report concerns a study of the distribution of starch in a typical refinery employing a vegetable carbon process. The fact that sugar crystals adsorb starch during their growth demonstrates the

difficulties involved in obtaining chemically pure sucrose by simple crystallization from water solutions. A practical example of difficulties from starch and other impurities is that of floc formation in carbonated beverages. In refining raw sugar by a vegetable carbon process, more than 90 percent of the starch which accompanies it is accounted for in the waste products of the operation. Granulated sugar contains about 7 percent of the starch originally introduced.

1318. SOME CHARACTERISTICS OF LOUISIANA RAW SUGARS

Balch, R. T.; and Friloux, J. J.

Sugar Bull. 34(17): 254-58; 34(18): 274-76. 1956

Studies were undertaken to determine causes for deterioration in the refining qualities of Louisiana raw sugars. None of the new varieties introduced was found to be refractory when properly prepared for milling. Mechanical harvesting, which has replaced hand-harvesting, and firing in place of hand-stripping, does not yield cane as clean and well prepared for milling as the old methods. Gummy fermentation during warm, wet weather, further increases processing difficulties, and continuous processing equipment adversely affected quality, especially during extended periods of wet weather. Characteristics studied during the 1951-54 seasons were the inorganic phosphate, starch, wax, and mineral content, sediment, and color.

1403. UNDETERMINED SUGAR LOSSES IN SUGARCANE FACTORIES:

CAUSES, DETECTION AND PREVENTION

Guilbeau, W. F.; and Martin, L. F.

Sugar J. 19(5): 36-38. 1956

In Louisiana sugarcane factories sugar is being lost, carried away in waste water, and is causing stream pollution. These losses can be located and determined with simple tests and apparatus. Installation of properly designed equipment will eliminate some of the entrainment losses. Good housekeeping and supervision will reduce to a minimum losses caused by micro-organisms. This could mean a saving of a very large amount of money annually to the industry.

1317. RESUME OF MILLING AND PROCESSING CHARACTERISTICS OF THREE NEW SUGARCANE VARIETIES

Guilbeau, W. F.; and Stewart, C. W.

Sugar Bull. 34(18): 276-277. 1956

Sugarcane variety C.P. 44/101 now comprises 50 percent of the total production in Louisiana. It was used as the standard of comparison in 1952, 1953, 1954, and 1955 for the evaluation of three new varieties. These are N. Co. 310, C.P. 48/103, and C. P. 47/193. Processing characteristics of each variety each year are detailed in tabular form; comparative ratings for each factor relative to the standard commercial variety are also given.

1319. THE VALUE OF FRESHER SUGARCANE TO BOTH GROWERS AND PROCESSORS  
Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.  
Sugar Bull. 34(14): 205-12. 1956.

An additional return of at least \$8 per acre for each day saved in getting cane to the mill after harvest can be expected by growers, according to results of the experiments reported. Average losses of recoverable sugar amounted to 15 percent five days after cutting, and almost 22 percent in about eight and one-half days. The tests were conducted during the 1954 and 1955 seasons; weather conditions and methods of handling were taken into consideration. Details of the experiments are given in tabulations of clarification conditions and processing results; lime requirements and mud quantities; and sugar recovery from cane; monetary returns are also tabulated for cane processed after two, three, seven, and eleven days. The largest gain can be realized only by grinding unburned, clean, fresh cane such as the combination harvester-cleaner-loader can deliver within 24 hours.

1400. PROCESSING CHARACTERISTICS OF THREE RECENTLY RELEASED SUGARCANES  
Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.  
Sugar J. 19(2): 37-40. 1956.

Experiments were completed in 1955 for the sixth consecutive season during which the processing characteristics of new sugarcanes have been determined by milling and clarification on a pilot-plant scale, in co-operation with the American Sugar Cane League and the Louisiana State University. Two new varieties released in 1955, C.P. 48/103 and C.P. 47/193, and N. Co. 310, released in 1954, were compared with the standard C.P. 44/101. Clarification characteristics of the three new canes and the standard are presented in data on mud quantities, clarities, purities, and the purity rises in clarification for both plant and stubble canes. Results for four consecutive seasons are summarized.

1246. EFFECT OF DELAY IN GRINDING ON VALUE AND PROCESSING QUALITY OF SUGARCANE JUICE.  
Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.  
Sugar J. 18(2): 28, 30-31. 1955.

Magnitude of the loss resulting from delay in delivery of harvested cane to the mills has been determined by experiments on the pilot-plant milling and processing of 2-ton samples taken at intervals of one, three, nine, and 14 days from six different large lots of cane kept in heap rows. Results indicate loss to the grower is substantially greater than in the factory, as most of the decrease in recoverable sugar takes place before the cane is purchased. In one extreme case, calculations show that the monetary loss to the grower would be as much as \$17.68 per acre for a 25-ton per acre stand of cane. The major loss incurred by the factories as a result of processing old cane with a low calculated recoverable sugar content arises from the sharp increase in overhead and unit

operating costs when actual production is less than 150 to 160 pounds of raw sugar per ton of cane.

1248. PROCESSING CHARACTERISTICS OF SUGARCANE VARIETIES GROWN IN 1954.

Guilbeau, W. F.; Coll, E. E.; and Martin, L. F.  
Sugar J. 18(6): 26-29. 1955.

Four varieties of sugarcane were tested for processing qualities, using the Standard C.P. 44/101 for comparison. The nature of determinations of mud quantities, clarities and other quantitative measurements made in continuous clarification on a pilot-plant scale are such that only gross differences in behavior can be established with reasonable accuracy but these differences are of greatest practical importance in sugarcane processing operations. This year C.P. 48/103 rated best of the varieties tested. N. Co. 310, next to C.P. 48/103 in sucrose and purity, produced a greater quantity of mud than the Standard C.P. 44/101, and clarity of juice was the lowest of all varieties. C.P. 47/193 ranked third in purity, with clarity equal to the standard; the quantity of mud produced was the largest of any variety, being 22 percent greater than the Standard C.P. 44/101. C.P. 44/154 was also tested. Tests were carried out in cooperation with the American Sugar Cane League, and under contract with the Louisiana State University.

1247. EVALUATION OF SUGARCANE VARIETIES C.P. 48/103 AND C.P. 47/193 FOR MILLING AND PROCESSING.

Stewart, C. W.; and Guilbeau, W. F.  
Sugar Bull. 33(2): 286-87. 1955.

Two varieties of sugarcane, C.P. 48/103 and C.P. 47/193, released for commercial use in 1955, were tested for milling and processing characteristics in 1952, 1953, and 1954. Results of these tests are given, and the two varieties were also rated against Standard Variety C.P. 44/101, milled under similar conditions.

1118. EVALUATION OF SUGARCANE VARIETY N. CO. 310 FOR MILLING AND PROCESSING

Stewart, C. W.; and Guilbeau, W. F.  
Sugar Bull. 32(19): 295. 1954.

Four examples of N. Co. 310 variety of sugarcane in 1952-53 grinding season and four samples in the 1953-54 season were evaluated in milling and juice-processing experiments. Average results showed a grinding rate and juice-processing performance slightly better than those of a standard variety. The purity of N. Co. 310 juice was higher, and the indicated theoretically available sugar per ton of cane was 107.0 percent of that of the standard cane.

## 1042. EVALUATION OF SUGAR CANE VARIETIES. A STUDY OF MILLING CHARACTERISTICS

PART I. Summary of Results - For Seasons 1950 and 1951.  
Schaffer, F. C.; Goodell, E. R.; Stewart, C. W.; and  
Keller, A. G.

PART II. Summary of Results - 1952.

Harlan, D.; Kunberger, A.; and Stewart, C. W.  
Eng. Expt. Sta. Bull. No. 42. Dept. Chem. Eng.  
Louisiana State Univ. 1954.

Determination of milling qualities of a sugarcane variety before it is released for commercial planting can help eliminate canes that might later cause processing difficulties. This advance evaluation of manufacturing qualities can best be obtained by grinding, experimentally, small lots (several tons) of the cane, under conditions simulating commercial practice, and then processing the juice. Milling tests were conducted on certain unreleased and commercial varieties of cane, using the facilities and staff of the Audubon Sugar Factory of Louisiana State University, during the 1950-51, 1951-52 and 1952-53 seasons. In these reports data are tabulated including average mill results on all tests made; and comparative rating of processing characteristics of the varieties processed each season. Bagasse densities are given for the 1952 season. C.P. 44/155 was promising for good factory performance in all three seasons. Because of the limited number of tests possible on the new varieties, the results should not be considered conclusive.

1116. SUGAR CANE PROCESSING. CLARIFICATION OF NEW VARIETIES OF CANE ON A PILOT-PLANT SCALE  
Guilbeau, W. F.; Lipps, J. G., Jr.; and Martin, L.F.  
Agr. Food Chem. 2: 941-46. 1954.

This research provides data on the suitability of new sugarcane varieties for sugar production before they are grown on a commercial scale. Equipment and procedures were developed for grinding and processing individual 2-ton samples of cane on a pilot-plant scale. The study of clarification of seven new varieties and a widely grown commercial standard on this scale provided information on the relative quantities of precipitate formed, filterability of the precipitates, and clarities of the juices. Significant differences in the performance of these new varieties were established, and were considered together with the results of agronomic field testing in deciding to release two of them for commercial use. Further field testing of one of the less promising new varieties was discontinued when it was established that juice from this cane would be extremely difficult to clarify.

## 1113. THE STARCH CONTENT OF SUGARCANE PRODUCTS: 1952 and 1953

## SEASONS

Balch, R. T.; and Friloux, J. J.

Sugar J. 17(5): 22-24. 1954.

The starch content of sugarcane is closely related to the physiological activity of the plant and is thus affected by conditions which govern this activity; starch gradually disappears from cane whose synthesizing powers are depressed or largely destroyed, as by freezing injury. Varieties exhibit different characteristics in respect to their starch content; the present commercial varieties grown in Louisiana contain a medium amount of starch. Starch is not eliminated in defecating juices by Louisiana practices. From 15 to 25 percent of the starch in sirup is taken up by the sugar crystals during their growth. The ratio of starch in washed raw sugar to that found in the original sugar was 0.81, showing that affirmation removes mostly that portion which is contained in the molasses film adhering to the crystals.

## 1117. SUGAR YIELDS, SUGAR RECOVERIES, AND THE FUTURE OF

## OUR LOUISIANA INDUSTRY

Martin, L. F.; Guilbeau, W. F.; and Lipps, J. G., Jr.

Sugar Bull. 32(22): 371-73. 1954.

There is the steadily increasing difference between yields and recoveries of sugar. There has been no significant increase or decrease in the pounds of sugar available per ton of cane, yet the recoveries of 96° raw sugar per ton of cane have continued generally downward. The sugar that is not recovered is being lost in increasing amounts in molasses, in bagasse, and in press cake. If the present trend should continue we can expect that only 70 percent of the available sugar will be recovered in 1956, rather than 80 percent, which was recovered in 1939. One line of work with the objective of remedying the situation is the evaluation of new varieties on a pilot-plant scale to ensure the introduction of only such new canes as are known to have good milling, clarification, and processing characteristics, at least when they are delivered to the factories in reasonably clean and fresh condition.

## 894. A PILOT PLANT FOR PROCESSING SMALL SAMPLES OF SUGARCANE

Guilbeau, W. F.; and Martin, L.F.

Sugar J. 16(1): 12, 14-15. 1953.

This small-scale pilot plant designed in consultation with the Department of Chemical Engineering of Louisiana State University and installed by the Bureau of Agricultural and Industrial Chemistry at the Audubon Sugar Factory has given satisfactory results in processing research during the past three grinding seasons. The small-scale plant permits experiments on cane and juice of uniform composition--at an average rate of only 50 gallons per hour, but continuously and simulating conditions in large-scale manufacture. An experiment can be completed in a day, using a 2- or 3- ton sample. Described are the

equipment and operations for juice extraction and storage, liming and heating, and evaporation, and requirements for installation and application.

759. A SECOND SEASON OF PILOT-PLANT PROCESSING OF SUGARCANE  
Guilbeau, W. F.; Black, C.L.; and Martin, L. F.  
Sugar J. 15(1): 15-17, 20, 34-36. 1952.

Twenty-six samples, representing ten different varieties of cane, were processed in the pilot plant during the 1951 grinding campaign. Five varieties were tested as stubble cane during the early part of the season and later as plant cane. A much larger number of experiments on replicated samples would be necessary to establish the significance of minor differences; but major differences in behavior can be determined with sufficient accuracy to provide information on what may be expected in commercial processing of the canes tested. The larger volume of mud and lower processing rates to be expected from certain varieties, as compared to others, can be checked consistently, and was generally the same this season as last.

825. ANALYTICAL STUDIES OF SUGARCANE JUICES PROCESSED ON A PILOT-PLANT SCALE.  
Fort, C. A.; and Smith, B. A.  
Sugar J. 15(7): 34-39. 1952.

This report covers data obtained during the 1950 and 1951 campaigns on the sugar and nonsugar composition of the raw juices and clarified juices of selected sugarcane varieties. The clarified juices were produced in pilot-plant operations. Subjects reported on are: The importance of suspended material in raw juice in mud production and rise in purity on clarification; elimination of phosphate as related to clarity; the clarification changes in lime, magnesia, and nitrogen; the composition of clarification muds; and the occurrence of unidentified organic nonsugars.

669. A STUDY OF THE PROCESSING OF SUGARCANE ON A PILOT-PLANT SCALE.  
Guilbeau, W. F.; Black, C. L.; and Martin, L. F.  
Sugar J. 14(6): 18-26, 28, 30. 1951.

The milling properties of samples of 8 varieties of sugarcane grown in the Baton Rouge area were determined at the Louisiana State University, and the samples were then ground in the University's Audubon Sugar Factory. The resulting juice was delivered to a pilot plant designed for complete continuous processing on small scale for studies of processing qualities of the juices, including the rate of clarification, the volume of mud produced, the clarity of the juice, and the increases in apparent purity effected by clarification under uniform conditions. The work established that small samples of cane can be used and continuous processing carried out on 250-300 gallons of juice to provide reproducible

data on the behavior of the juice in clarification which is applicable to factory operations. This makes possible the relatively early evaluation in this respect of new varieties of which only limited quantities are available.

598. A STUDY OF THE PROCESSING OF SUGARCANE ON A PILOT-PLANT SCALE

Guilbeau, W. F.; Black, C. L.; and Martin, L. F.  
(Condensed version) Sugar Bul. 29(14): 214-19. 1951.

Samples of 8 varieties of cane were ground in the Audubon Sugar Factory Mill and the juice was delivered to the processing pilot plant. Results are given on the relative processing qualities of the samples tested, showing that rates of clarification varied from about 30 to 60 gallons per hour, dependent largely upon the clarification mud weights. The clarities and increases in purity effected were not so consistent as the processing rates, but their relative values followed the trend of other qualities of the varieties studied. A severe freeze on November 25 resulting in the killing of the standing canes had a distinct effect on processing results in the experiments made from that time until the end of the 1950 grinding season. It has been established that small samples of cane can be used and continuous processing carried out on 250-300 gallons of juice to provide reproducible data which is applicable to factory operations.

473.3 EFFECT OF BURNING ON THE DETERIORATION OF SUGARCANE  
UNDER LOUISIANA CONDITIONS

Balch, R. T.; Broeg, C. B.; and Lauritzen, J. I.  
Tech. Bull. 1021. 1950.

In order to obtain more basic information on the effect of burning on mill cane, especially under Louisiana conditions, so as to evaluate better the losses which are brought about by delayed milling, a rather detailed study of the subject was undertaken by the authors. This paper deals with only one phase of the problem, namely, the relative effects of moisture and temperature on the deterioration characteristics of burned cane in comparison to those of unburned cane stored under the same conditions. The data are not translatable into terms of field practices, but it is expected that further work will supply this information.

325.22 POWDERED CALCIUM CARBONATE (OYSTER SHELL FLOUR) IN  
PROCESSING OF SUGARCANE

Ventre, E. K.: and Balch, R. T.  
U. S. Dept. Agr., Bur. Agr. Ind. Chem., AIC-252. 1949.  
Processed.  
Sugar Bull. 28(2): 22-3, 27-9. 1949.  
Sugar J. 12(6): 10-11. 1949.

As a result of laboratory experiments on clarification of refractory juices, powdered calcium carbonate in the form of oyster shell flour

was used in varying quantities by a number of factories during the 1948-49 season. The majority opinion was that this material had definite merit. The few differing opinions, it is believed, arose from the manner of using the calcium carbonate, since the urgency of the need for a clarification aid had not permitted preliminary experimentation and checking. Presented here are specific recommendations which should give satisfactory results if carefully followed.

325.23 EFFECT OF FREEZING TEMPERATURES ON DIFFERENT VARIETIES OF SUGARCANE AND THE MILLABILITY OF DAMAGED SUGARCANE IN LOUISIANA

Lauritzen, John I.; Balch, R. T.; Davidson, Lester G.; and Arceneaux, George.

Technical Bull. 991. 1949.

Extensive observations were made of freeze injury of a number of different varieties of sugarcane in commercial fields and experimental plots. Varieties subjected to different levels of freezing conditions were compared with respect to the normal and deleterious chemical and physiological changes occurring in standing and windrowed cane and in cane stored at different temperatures. Sharp differences in varieties with respect to symptoms and deleterious changes were not found. Minor differences were not always consistent at different levels of freezing conditions. Badly frozen cane kept better in the windrow than when standing and at lower than at higher temperatures. The results show that cane in which a rather high percentage of leaves and terminal buds are injured or killed, and in which a limited percentage of eyes are injured or killed by freezing temperatures, may under favorable conditions synthesize sucrose.

325.24 LOSSES RESULTING FROM DELAYED MILLING OF SUGARCANE

Balch, R. T.; Broeg, C. B.; and Lauritzen, J. I.

Sugar Bull. 27(3): 36, 45-7. 1948.

Decreased yields of cane and sugar from the Louisiana crops led to the study here reported on weight losses and sucrose and quality losses that result from delayed milling of sugarcane. This study is presented as an incentive to the industry to investigate harvesting methods in detail and thus lead to greatly improved field-harvesting practices. Deterioration characteristics of sound unburned mill cane are reviewed briefly including a table showing the relative inversion rating of commercial sugarcanes. Burned cane and its deterioration problems are also discussed. Conclusions are drawn that burning cut cane for trash removal creates serious deterioration problems if milling is delayed appreciably. Evidence is presented that every effort should be made to mill harvested, burned cane within two days after cutting during the early part of the season (with a somewhat longer lag in the latter part tolerated). The solution suggested lies in prompt milling of cut cane, both burned and unburned.

261.1 THE HOUMA, LOUISIANA, STATION OF THE BUREAU OF  
AGRICULTURE AND INDUSTRIAL CHEMISTRY,  
U. S. DEPARTMENT OF AGRICULTURE  
Balch, R. T.  
Sugar J. 9(10): 8-10. 1947

The increasing number of new and relatively unknown varieties of sugarcane for commercial planting in Louisiana in the late 1920's caused processors to request the aid of Government chemists in obtaining information on the composition and behavior of cane juices as to sirup and sugar production. Fear that undesirable varieties might be extensively planted ahead of the technical knowledge of the workability of the juices led to the establishment of a field station to study the chemical and technological phases of sirup and sugar production. The establishment and operation of the Houma Station are described as well as the work done there to further basic knowledge of the chemistry of sugarcane.

325.25 CLARIFICATION OF LOUISIANA CANE JUICES AND RELATED  
PROBLEMS  
Balch, R. T.  
Proc. of Louisiana Sugar Cane Technologists Assoc.  
Oct. 3, 1945  
Sugar Bull. 24(4): 28. 1945.  
(No reprints)

The most urgent problems in clarification of Louisiana cane juices are: (1) an evaluation of the newly released and of the most promising of the unreleased varieties of sugarcane from the standpoint of standard clarification methods; (2) and evaluation of the effects of trash from the standpoints of clarification and/or recovery of sugar; and (3) the necessity to develop more efficient methods of clarification.

325.26 FARM PRODUCTION OF SUGARCANE SIRUP  
Walton, C.F., Jr.; Ventre, E.K.; McCalip, M.A.; and Fort, C.A.  
Farmers' Bull. 1874. 1941.  
(No reprints)

The use of sugarcane for sirup is fully discussed with all the facets of the production of sugarcane sirup in the United States. Contents are divided to treat the composition of sugarcane juice, harvesting problems, extracting, treating and evaporating the juice, canning and marketing problems, the cost, composition and food value as well as the byproducts. This bulletin describes recent improvements in the procedures recommended for making sirup of better and more uniform quality. The equipment and methods described are those most practicable for small farm or community plants.

325.27 WINDROWING AND STORING OF SUGARCANE IN LOUISIANA  
 FOLLOWING INJURY BY FREEZING TEMPERATURES  
 Lauritzen, J.I.; Fort, C.A.; and Balch, R.T.  
 U. S. Dept. Agr. Tech. Bull. 738, 44 pp. 1940.

The data presented deal primarily with experiments of windrowing and storage of cane of varieties Co. 281 and Co. 290 injured by freezing temperatures. They justify the conclusion that it is safer, when practical, to windrow cane before it has been injured by low temperatures. They also justify the windrowing of damaged cane in which sound eyes are found as a protection against further injury.

BYPRODUCTS

2044. DEVELOPMENTS IN THE EXTRACTION OF SUGARCANE WAX  
 Pominski, J.; and Kopacz, B.M.  
 Brit. Chem. Eng. 5: 860-62. 1960.

It has been shown on a laboratory scale that the principles of filtration-extraction can be successfully applied to sugarcane mud to produce crude sugarcane wax. Sugarcane muds subjected to this process have mass velocities as high as 5900 pounds/hour/square foot and yield crude sugarcane waxes with a high efficiency of recovery. The small scale experiments indicate the need for further work on a large scale.

1630. THE ECONOMIC BASIS FOR UTILIZATION OF BAGASSE  
 Martin, L.F.  
 Sugar y Azucar 53(6): 32-33, 38 (English);  
 52-54, 70 (Spanish). 1958.

Bagasse is used to make insulating board, paper, chemical pulp, plastics furfural, and as a feed ingredient in at least some sugarcane-growing areas. If these uses are economically sound, why is not all available bagasse utilized profitably? The explanation is that availability and economics are relative and variable factors. Availability varies from 100 percent to a deficit, depending upon replacement fuels in different localities. Economic considerations differ in each area, and even between factories in an area. The framework within which existing or proposed uses must be considered includes: (1) Cost of alternative fuel, (2) mill and factory efficiency and surplus actually available, if any, (3) scale of utilization, nature, and location of processing facility, (4) cost of collection, storage, and handling, if not converted at the sugar factory during the production season, (5) chemical and operating costs of processing, and (6) markets for the end products. General factors and relationships are given, to which the authoritative discussions of successful uses of bagasse in the symposium may be related. They will be most useful in considering extension of established uses in new areas, and especially for evaluating any new uses of bagasse that may be developed.

## 1547. EXTRACTION OF WAX FROM U. S. SUGARCANE MUD

Pominski, Joseph; Spadaro, J. J.; and Vix, H. L. E.  
Sugar J. 20(2): 17 and 38. 1957.

It has been shown that the principles of filtration-extraction can be successfully applied to sugarcane mud on a laboratory scale to produce crude sugarcane wax. Sugarcane muds subjected to this process have mass velocities as high as 5900 pounds/hour/square foot and yield crude sugarcane waxes with a high efficiency of recovery.

## 1192. RECOVERING ORGANIC ACIDS FROM FERMENTATION WASTES BY IRON SALT PRECIPITATION

Fort, C. A.; and Smith, B. A.  
U. S. Patent No. 2,711,425, June 21, 1955.

A process for the production of aconitic acid comprising adjusting the pH of the waste derived from the fermentation of a cane sugar bearing material to about from 3.5 to 4.5, mixing a water soluble iron salt with the waste, isolating the precipitate of iron salt of aconitic salt formed in the acidified waste, dissolving the precipitate in an aqueous inorganic acid, and isolating the aconitic acid so produced.

## 1177. PROGRESS IN THE DEVELOPMENT OF BYPRODUCTS OF SUGARCANE IN LOUISIANA

Martin, L. F.  
Sugar J. 17(10): 14, 15, 37, 39. 1955.

Development of sugarcane byproducts in Louisiana since 1951 is discussed. Manufacture of insulating board from bagasse is being expanded, and paper is also being commercially manufactured, with furfural and high grade chemical cellulose manufacture as possibilities. Developments in molasses, sugarcane wax and aconitate from molasses offer commercial prospects. Increased recovery of sugar at low cost is afforded by ion-exchange purification to produce direct-consumption sugar.

## 1115. ACONITIC AND TRICARBALLYLIC ACID ESTERS AS VINYL PLASTICIZERS

Roberts, E. J.; Martin, L. F.; Magne, F. C.; and Mod, R.R.  
Rubber World 130: 801-804. 1954.

Esters of aconitic acid and mixed esters of the corresponding tricarballylates obtained by hydrogenation of the aconitic acid esters have been prepared using alcohols ranging from propyl to octyl. These esters have been screened for use as plasticizers for polyvinyl chloride acetate plastic, and the results indicate that some of them may be used in place of di-2-ethyl hexyl phthalate plasticizer.

967. FATE OF SOME NONVOLATILE ORGANIC ACIDS OF SUGARCANE JUICE IN FERMENTATION  
Roberts, E. J.; Fort, C. A.; and Martin, L. F.  
Sugar J. 16(6): 36-37. 1953.

Stillage and the molasses from which it was produced were examined by accurate analytical and chromatographic methods. Commercial stillage produced from molasses containing 3.20 percent aconitic acid on solids, contained 7.97 percent aconitic acid on a solids basis. From the relative weights of solids before and after fermentation, it was calculated that 85.3 percent of the original amount of acid was present in the stillage. In a laboratory fermentation during 111 hours, 82 percent of the aconitic acid was recovered. Acids separated from the cane juice and molasses fermentation stillage were identified as mesaconic, fumaric, succinic, aconitic, glycollic, malic, citric, and oxalic acids. This is the first time that any of these acids other than aconitic has been isolated from sugarcane products and completely identified by the preparation of derivatives.

907. PLASTICIZERS FROM ACONITIC AND TRICARBALLYLIC ACIDS.  
Magne, F. C.; and Mod, R. R.  
Eng. Chem. 45: 1546-47. 1953.

Rapid expansion in the field of vinyl resin plasticizers has increased the demand for efficient plasticizing materials. Since aconitic and tricarballylic acids have three esterifiable groups, their esters are potential plasticizers for many resins. In screening tests most of the esters equalled and some excelled dioctyl phthalate in the various plasticizing properties (tensile strength, modulus at 100% elongation, and brittle temperature), and in vapor pressure, density, and viscosity. The results suggest that most of these esters may be substituted for dioctyl phthalate in plasticizing vinyl resin.

826. ACONITIC ACID CONTENT AND COMPOSITION OF LOUISIANA BLACKSTRAP MOLASSES  
Fort, C. A.; Smith, B. A.; Black, C. L.; and Martin, L. F.  
Sugar 47(10): 33-35. 1952.

This study provides for the first time a reliable estimate of the total amount of aconitic acid available from an annual cane crop. More representative and accurate information on molasses composition has been obtained than has been available from earlier analyses. It is feasible by present methods to recover about one-third of the aconitic acid as calcium-magnesium aconitate, providing 9 to 11 million pounds of the salt, or 4 1/2 to 5 1/2 million pounds of equivalent aconitic acid per year. Relations of the inorganic constituents to sugars, to total organic nonsugars, and to aconitic acid in particular, have been determined for the three different areas of the Louisiana sugar belt. Data are presented on a true solids basis permitting more accurate comparisons than are possible from apparent percentages based on Brix, customarily used in the industry.

761. MOLASSES, IMPORTANT BUT NEGLECTED PRODUCT OF SUGARCANE  
 Wolfrom, M.L.; Binkley, W.W.; and Martin, L.F.  
 Sugar 47(5): 33-35. 1952.  
 Internat'l. Sugar J. 54(645): 249-50. 1952.

About 15 percent of the constituents of molasses are yet unknown. The application of modern analytical techniques, such as chromatography, in exploring this field is described, and some findings of a study of molasses composition (under a Research and Marketing Act contract with Ohio State University) are reported. The importance of the complex, highly polymerized, and yet uncharacterized substances in affecting every step in the manufacture of cane sugar, and in the development of new uses for molasses, is pointed out.

599. UTILIZATION OF BYPRODUCTS OF LOUISIANA SUGARCANE  
 Martin, L.F.  
 Sugar J. 13(12): 26, 27, 46, 48, 50, 52. 1951.

The broader aspects of the commercial processes utilizing byproducts of the Louisiana cane-sugar industry are reviewed briefly. References are given to the most comprehensive publications on the history and technology of the development of these byproduct industries. These include the production of insulating board, molding plastic, bagasse mulch and litter, dried bagasse-molasses feed, and aconitic acid. The economic importance of these developments to the industry is pointed out and possibilities for future developments are discussed briefly. While research continues on the apparently unlimited possibilities of recovering valuable byproducts in the process of cane-sugar manufacture in Louisiana, the list of established accomplishments is impressive. Among developments for the immediate future are processes for more complete recovery of the large potential supply of aconitic acid, and new approaches to the problem of economically separating bagasse into fractions more suitable for specific applications in feedstuffs and in high-grade papermaking.

325.11 WAX AND FATTY BYPRODUCTS FROM SUGARCANE  
 Sugar Research Foundation, Inc. Technological  
 Report Series No. 3. 1947.  
 325.12 WAXES AND FATS FROM SUGARCANE  
 Chemurgic Dig., 7(7): 11-15. 1948. (No reprints)  
 Balch, R.T.

It has been estimated that the cane-sugar factories of the U. S., Puerto Rico and Cuba throw away about 60 million pounds of crude sugar-cane wax annually. From this amount of crude wax about 38 million pounds of a usable dark hard wax and 22 million pounds of fatty material of as yet uncertain value might be obtained. Solvent-extraction and separation methods appear to be the best approach to the problem of producing a uniform hard wax. The crude wax is extractable from press cake by a number of solvents, but a close-cut petroleum naphtha is suggested for commercial use. The crude wax can

be resolved into its hard-wax and fatty-fractions by direct crystallization of the wax from solution or by extraction of the fatty fraction with a selective solvent. Methods of extraction are described and cost of extracting crude wax from press cake and resolving it into its hard-wax and fatty components at certain production levels is given (now obsolete).

325.13 SUGARCANE WAX STUDIES, 1942-43

Balch, R. T.; and Broeg, C. B.

Sugar Bull. 22(14): 106-9; 22(15): 117-19; 22(16): 123-25, 127. 1944. (No reprints).

Sugarcane (variety Co. 290) has a crude-wax content of 0.2 percent and, based on this and other results with different varieties, there is a potential yield of about 18 million pounds of crude wax in Louisiana from an average cane crop. Petroleum naphthas (bp 85 to 120°) can be successfully used for extraction of the wax from the press-mud. A cane wax having a color equal to that of No. I carnauba wax was obtained by treating an alcohol solution with decolorizing carbon.

325.14 SUGARCANE WAX STUDIES, 1941-42

Balch, R. T.; and Broeg, C. B.

Sugar Bull. 20: 189-91. 1942. (No reprints)

A resume is presented of the findings relative to the possibility of recovering sugarcane wax and other byproducts from the sugarcane industry, with a description of the two main lines on which the investigations on cane wax were conducted. Distribution of crude wax in sugar factory products and pilot-plant wax extraction studies are discussed. Tables give the distribution of crude wax in sugar factory products as well as the wax and fats content of mud press-cakes and solvent extraction of crude wax.

325.15 PRELIMINARY REPORT ON SUGARCANE WAX

Balch, R. T.

U. S. Dept. Agr., Agr. Chem. Eng., ACE-99. (Processed). 1941.

Sugarcane wax was first used industrially in Natal, South Africa, during World War I. The need among wax users for a domestic source of wax to supplement the supply of imported carnauba prompted the study of sugarcane wax by our Bureau. This paper discusses step by step the initial studies undertaken on the extraction of wax from mud press-cake and describes methods for removing the fatty fraction. The process found most satisfactory is summarized by presenting a flow diagram which is based on laboratory experience. Tables show wax content of Louisiana mud press-cakes (moisture free basis) and solubility of cuticle wax in various solvents.

ION-EXCHANGE SUGARS

## 1476. EXPERIMENTS ON PURIFICATION OF PUERTO RICAN SUGARCANE JUICES

Smith, B. A.; Sanchez-Nieva, F.; Colon, L. F.; and Coll, E. F.

Sugar J. 19(12): 20, 21, 58, 59, 61. 1957.

Treatment of commercially clarified Puerto Rican sugarcane juices by a noninverting reverse cycle arrangement of ion-exchange resins produced effluent juices from which high quality white sugars were crystallized. Subsequent experiments using the same anion and cation resins in a "mixed bed" provided juices yielding sugars of inferior color and turbidity. The quantities of chemicals required for regeneration of the exchanger resins in the mixed bed were greater than when the resins were in separate columns.

## 1401. PURIFICATION OF CLARIFIED SUGARCANE JUICE AND

QUALITY OF SUGAR PRODUCED BY ION-EXCHANGE

Smith, B. A.; Fort, C. A.; and Martin, L. F.

Proc. Tech. Sess. Bone Char, 1956, 267-80

Experiments with juices reconstituted from clarified juice sirups have been previously reported, in which suitable anion and cation exchangers were compared for removal of ash, nitrogen, and color. Subsequent work forming the basis of this report was carried out with freshly clarified juices purified by ion-exchange before they were concentrated to sirups. Exchangers having the highest capacities for removing anions and cations, together with desirable properties for removing nitrogen and color, were employed for most of the experiments. Alternative anion exchangers were tried as "scavengers" to remove acidity liberated by the cation exchanger, and were compared particularly with regard to nitrogen and color removal. Data presented are particularly those on effective elimination of specific nonsugars, changes in composition of juices and sirups by variations of the ion-exchange treatment, and residual nonsugars affecting the quality of sugar obtainable by crystallization without bone char or carbon decolorization.

## 1176. ION-EXCHANGE: QUALITY OF SUGAR PRODUCED BY REVERSE CYCLE PURIFICATION OF JUICES

Fort, C. A.; and Smith, B. A.

Sugar 50(4): 43-45. 1955.

Work was carried out with freshly clarified juices purified by ion-exchange before being concentrated to sirups. Experiments were on a scale sufficiently large to produce enough sirup for sugar boiling and to provide information on quality of the sugar. Exchangers having the highest capacities for removing anions and cations, together with desirable properties for removing nitrogen and color,

were employed for most of the experiment. Alternative anion exchangers were tried as "scavengers" to remove acidity liberated by the cation exchanger, and were compared particularly with regard to nitrogen and color removal. Data are presented on effective elimination of specific nonsugars, changes in composition of juices and sirups by variations of the ion-exchange treatment, and residual nonsugars affecting the quality of sugar obtainable by crystallization without bone char or carbon decolorization.

1041. COMPARISON OF THE EFFECTIVENESS OF SELECTED ION-EXCHANGE RESINS FOR THE PURIFICATION OF CLARIFIED SUGARCANE JUICE  
Fort, C. A.; Smith, B. A.; and Martin, L. F.  
Proc. Third Technical Session on Bone Char, 179-98. 1953.

Properties and capacities of anion-exchangers of the salt-splitting type, and three cation exchange resins for removing ionic impurities and color from sugarcane juice have been determined individually, in experiments on the purification of reconstituted juice which had been evaporated to sirup density for storage, as well as by a combination of the selected anion- and cation-exchangers, followed by treatment by weakly basic or decolorizing resins for neutralization and color removal. Results obtained by means of reverse cycle purification were compared with those obtained by treatment of the same natural and synthetic model juices with a strong anion-exchanger followed by a strong cation-exchanger, and also with the results of conventional demineralization using a strong - cation-exchanger first, followed by a weakly basic anion-exchanger.

893. REVERSE CYCLE DEMINERALIZATION OF SUGARCANE JUICES  
WITH ION-EXCHANGE RESINS  
Fort, C. A.; and Smith, B. A.  
Sugar J. 15(9): 16, 18, 22-25. 1953.

Clarified sugarcane juices and synthetic model juices have been ion-exchanged by the reverse demineralization cycle, with negligible inversion of sucrose. When a strongly acidic action-exchanger was substituted in this cycle, it caused a 2.8 percent inversion of the sucrose present, while direct demineralization, using such a cation-exchanger as the first element in the cycle, gave a 4 percent inversion. Other points discussed include need for using dry solids and true sucrose for correct sugar and nonsugar balances; effect of removing reducing substances on the sugar balances; need for using batch operation of ion-exchange to correctly determine the inversion of sucrose; effect of nonsugar composition on purity rise by ion-exchange; the presence of neutral organic nonsugars in juices which are of unknown composition.

CONFECTIONERY

## PROGRESS IN CANDY RESEARCH

Authors, various.

Reports Nos. 1 through 32.

1940-1958.

These reports describe candy investigations conducted at the Southern Utilization Research and Development Division in cooperation with the National Confectioners' Association. A wide variety of information on the ingredients, preparation and storage of different kinds of candy is presented. The Southern Division has no reprints available for distribution. Individual copies may be obtained by writing to the Council on Candy, National Confectioners' Association, One North LaSalle Street, Chicago 2, Illinois.

## 1631. CANDY TECHNOLOGY IN RETROSPECT AND IN PROSPECT

Martin, L. F.

Candy Ind. and Confectioners' J. 110(13): 82, 91-92. 1958.

A general information article reviewing advances in candy technology during the past 75 years. The paper discusses the need for expanded research on fundamental investigations and technology if the industry is to continue to make progress in the 75 years to come.

## 1314. CANDY BIBLIOGRAPHY

Robinson, H. M.; and Skau, D.B.

Processed. 154 pp. 1956.

This bibliography covers significant references in candy journals for the years 1944 to July 1954. Also included are a few references from earlier years and some references from other journals on chemical methods or theories applicable to the candy field. The 1,466 references in the bibliography are completely indexed in the subject index that follows the citations.

## 1402. STABILIZATION OF FATS IN CANDY. II. THE IMPORTANCE

OF FREE FATTY ACIDS

Robinson, Helen M.

Mfg. Confectioner 36(9): 21-23. 1956.

The value of adding various antioxidants and glycerol to the fats incorporated in candies has been investigated. The fats used included butter, hardened vegetable oil, commercial animal fat shortenings, and hardened coconut oil. Candies containing freshly churned butter had better keeping quality than those made with a high-score butter stored under refrigeration. Eleven antioxidants gave excellent protection against oxidative rancidity. The formation of free fatty acid was retarded by the addition of glycerol but not by the addition of antioxidants.

1320. MARTIN CALLS FOR MORE RESEARCH IN CANDY, SUMMARIZES  
RESULTS OF LIMITED STUDIES  
Martin, L. F.  
Confectionery and Ice Cream World 55(24): 34, 36. 1956.

Some of the results of 13 years of candy research are described, and the need for further research to solve some of the remaining problems of the candy industry are cited. Among the accomplishments mentioned are the use of fruit essences in candies; cranberry candies; value of brewer's yeast antioxidants; use of dry sweet whey, and calcium carbonate and turbinado sugar in candymaking; and several others. Areas in which further research is urgently needed are said to be keeping quality, such as tenderness and shelf life of starch jellies, moisture balance, and improvements in processing methods to produce better candies. It is pointed out that the direct loss from returned goods is \$5 to \$7 million per year.

1175. SUGAR IN CONFECTIONERY  
Martin, L. F.  
Advances in Chem. Ser. No. 12: 64-69. 1955.

This is a summary of problems of the candy industry, recent advances in applicable research, and some of the research still needed. Review of recent advances in explaining reactions of sugars in candymaking emphasizes the need for applying the methods and results of this research to the candymaker's art. Highly concentrated solutions of sugars at elevated temperatures undergo complex transformations, some initial products of which have been identified. Reactions of sugars with proteins have been the subject of extensive recent research. Formation of sugar anhydrides and caramelization products can account for many production difficulties and shortcomings of improperly processed hard candies. Caramels are an example of candies in which it is desirable to accelerate as well as control the complex degradation reaction of sugars with proteins.

1038. PEANUT BRITTLE RANCIDITY  
Anonymous  
Candy Res. Summ. No. 2  
U. S. Dept. Agr., Bur. Agr. Ind. Chem., AIC-368, 4 pp.  
1952. Processed

This Candy Research Summary presents conclusions reached in experiments being sponsored jointly by USDA and the National Confectioners' Association. Peanut brittle to which dairy butter has been added is the product studied. Antioxidants in the sugar part of the candy do not prevent rancidity in the peanuts. Peanut oil in peanut brittle becomes rancid after storage of 2-1/2 months at room temperature. This booklet tells how the development of rancidity can be minimized by use of fresh peanuts.

## 895. ANTIOXIDANTS

Anonymous

U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC-368.

6 pp. 1953. Processed.

A conclusion reached in studies of the value of antioxidants in stabilizing certain types of candy is that the right antioxidant, used properly, will prolong shelf-life. Explained are: when to use and when not to use antioxidants; which antioxidants to use; and how to prepare and add the antioxidant to the candy batch. Suppliers of the effective antioxidants are listed. The stable shelf-life found in experiments with 7 commercial antioxidants added to cream fondant with 5% butter is illustrated by a bar graph. (This is research supported jointly by BAIC and the National Confectioners' Association.)

## 827. RESEARCH CAN AID THE COST-PROFIT FIGHT.

Martin, L. F.

Western Confectioner 32(7): 11. Research Needed to Reduce Spoilage; Cut Costs; and Increase Production. Candy Ind. 17(2): 8,12,25. The Problem of Improving the Shelf-life of Candy -- Research Contributions Towards its Solution. Address, Natl. Confectioners' Assoc., Chicago, Ill., June 1-5. 1952.

Approaches towards solving the problem of losses up to 5 to 6 million dollars annually in the candy industry from returned goods due to poor storage quality, include proper choice of high-quality raw materials, improved formulas and production methods, and the addition of new ingredients. Examples are given of what can be accomplished through research along these lines.

## 597. A STUDY OF THE STABILIZATION OF FATS INCORPORATED IN CANDY

Robinson, Helen M.

Food Tech. 5(1): 20-24. 1951.

A method of accelerating fat oxidation by the use of traces of copper, added as copper stearate, has been applied in studying the deterioration of fats in candies. The method was applied to test samples of butter and of hardened vegetable oils for peroxide values and free fatty acids after storage at 86° F. in air ovens. The fats in butter creams and butter caramels were tested in the same way. A series of antioxidants was tested in the fats alone and in the fats with added copper, and with these same fats incorporated in candies. Hardened vegetable oils showed very little oxidative rancidity even with the added copper; so antioxidants were of little value. Butter with added copper showed definite peroxide values in a short period and this period can be materially lengthened by the use of antioxidants. Final evaluation of the results awaits the completion of the tests on candies without the added accelerator.

775.3 MILK SOLIDS GIVE CANDIES LONGER LASTING FRESHNESS  
Hall, H. H.; and Fahs, Fred J.  
Food Ind. 21: 1042-43. 1949.

In a study of seven kinds of dry milk solids as ingredients of candy, it was observed that they possess different moisture-retaining capacities in cream fondant, an indication of their possible importance in maintaining candy freshness and prolonging shelf-life. Although most dairy products are used by the candy industry for flavor and texture, storage tests of fondant containing milk solids showed that their moisture loss by evaporation was less than that of control pieces. Quantitative data is presented on the ability of the individual products to hold moisture in candy as an aid in selecting milk solids for this purpose. Storage experiments were carried out to obtain information on the value of powdered milk products as a group as moisture-retaining ingredients, the moisture-retaining values of different amounts of the products in candy, the differences between the moisture-retaining values of the individual products.

325.28 NEW INGREDIENTS FOR CANDY PRODUCTION  
Martin, L. F.  
Mnfg. Confec., 29(9): 17-18. 1949.

Food products utilized in candymaking cover the entire range of dairy products, fruits, nut meats, and others too numerous to list individually. The purpose of the research described here is to utilize farm products in the confectionery industry to the mutual advantage of farmers and candy manufacturers. Formulas and other helpful data regarding new ingredients that have been tried and found valuable, are given. A survey revealing candy preferences is reported.

792.1 METHODS AND RESULTS OF ANALYSIS AND EXAMINATION OF CANDY  
AND ITS INGREDIENTS  
A List of References  
Hall, H. H.  
U. S. Dept. Agr., Bur. Agr. Ind. Chem., AIC-182,  
9 pp. Processed. 1948.

A list of 68 references serves as a ready guide to this phase of the candy literature. The journal, Chemical Abstracts, served as the principal source of references and in such cases the abstract reference is given together with that of the original publication. No particular attempt is made to abstract each article other than to indicate something of the nature of its contents.

## 325.19 CANDY: ITS INGREDIENTS AND MANUFACTURE

## A List of References

Hall, H.H.

U. S. Dept. Agr., Bur. Agr. Ind. Chem., AIC-181,  
20 pp. 1948. Processed

This bibliography covers a large part of the technical literature relative to the chemistry and use of ingredients in the manufacture of candy. References are also given to literature pertaining to various phases of candy manufacture, such as physical and chemical measurements, process controls, storage, spoilage, etc. These literature references, with brief abstracts, are intended to serve as a guide to the several subjects. The journal, Chemical Abstracts, has served as the principal source of references and in such cases the abstract reference is given together with that of the original publication. Twenty pages, with index.

## 111.1 DRESSINGS FOR CANDY SLABS

Hall, H. H.; and Fahs, F. J.

Mfg. Confectioner 27(9): 40-41. 1947

A search has been undertaken for a replacement of mineral oil as a dressing for candy slabs. The properties required of a suitable slab dressing material are given as well as the methods which have been adopted for determining the suitability of a number of possible replacement products. Results are given for a number of vegetable oils on which tests have been completed. None of these vegetable oils is sufficiently stable to be used as a candy-slab dressing. Tests on other possible substitutes are in progress, or will be undertaken as new products become available.

## 659.4 IMPROVED FRUIT MARSHMALLOW

Hall, H. H.; and Fahs, Fred J.

Mfg. Confectioner 27(10): 32,35. 1947

(No Reprints)

The keeping quality and appearance of fruit marshmallow containing concentrated fruit puree was improved by the addition of the enzyme invertase to cause hydrolysis of sucrose, thus increasing the liquid phase of the candy. The structure of the marshmallow was strengthened by the addition of a small amount of pectin to form a very weak gel. The basic formula and methods for incorporating invertase and pectin are given together with observations on the keeping quality of marshmallows stored for 90 days, and the acceptability of the pieces by qualified taste testers.

## 775.2 ISOLATED PROTEINS IN CANDY MAKING - PART I

Hall, H. H.; and Fahs, Fred J.

Mfg. Confectioner 26(11): 27-28, 77.

1946.

Preliminary studies were made on the use of isolated peanut and soybean proteins as ingredients of candies. Peanut protein prepared in a pilot plant was found to contain residual peanut flavor which was imparted to candies. Soybean protein was incorporated into batches of honey-flavored, and vanilla-flavored nougats, hard candies, and cast creams, and the effects on the quality factors, color, texture, and flavor, were determined.

## 775.4 ISOLATED PROTEINS IN CANDY MAKING - PART II

Hall, H. H.; and Fahs, F. J.

Mfg. Confectioner 27(11): 32-35.

1947.

Methods have been developed for the incorporation of soybean proteins into representative types of hard candies. Although it is not possible to incorporate insoluble proteins into plain, clear, hard candies without causing clouding, the appearance is enhanced by placing a clear wrapper over the protein containing protein of the candy. The graining characteristics and other quality factors of white sugared mints are not changed by the addition of proteins. However, the results suggest the desirability of using almost white protein products for candy of this type. Formulas are given for experimental hard candies containing 10.73 percent and 7.23 percent protein.

## 325.20 NEW AGRICULTURAL PRODUCTS USED IN CANDY

Hall, H. H.; and Fahs, F. J.; and Charbonnet, Louise H.

Food Ind. 18: 1008-10, 1172.

1946.

Oilseed, cereal, and legume products, which are sources of protein, fat, carbohydrates, minerals, and vitamins, were incorporated into experimental candies. It was found that from four to seven percent of pulverized products derived from wheat, cottonseed, soybeans, and peanuts could be incorporated into nougat, caramel, and fudge without greatly changing the usual characteristics of these candies. Most of the experimental ingredients possessed flavors, which limited the amounts that could be incorporated into particular types of candy. Varying amounts of the vitamins, thiamin, riboflavin, and niacin were added to candies by the special ingredients. Assays made on stored candies showed that the thiamin and riboflavin were well retained for 180 days or longer at 74 to 80° F. and 55 percent relative humidity.

## 659.2 MODIFIED PECTINS MAKE POSSIBLE NEW TYPE CANDIES

Hall, H. H.; and Fahs, F. J.

The Confectioner 31(6): 10, 11, 37.

1946.

Formulas are given for the preparation of experimental batches of pectin jellies and a butterscotch cream-type center with low-methoxy

pectin as the gelling agent. Comparison of jellies made with low-methoxyl pectin and 150-grade pectin show a saving of 50 percent of sucrose. The sugar content of the butterscotch cream-type center is 12.9 percent, compared with 58.2 percent in ordinary cream fondant and 31.1 percent in centers made with 150-grade pectin.

863.1 REFERENCES ON CANDY MAKING AND SOURCES OF EQUIPMENT AND MATERIALS

Anonymous

U. S. Dept. Agr., Bur. Agr. Ind. Chem., AIC-112,  
5 pp. 1945. Processed

Since the Department of Agriculture has not issued publications relating to the preparation and handling of ingredients for candy making as commonly used by the industry, this circular was prepared to serve as a guide for prospective and established candymakers to sources of information. It contains lists of references to books and trade journals, together with the names of a number of manufacturers and suppliers of equipment and materials.

325.21 TESTING NEW INGREDIENTS IN CANDY AT REGIONAL LABORATORY

Hall, H. H.  
Intern. Confectioner 55(6): 34. 1945.

A partial report on recent developments at the Southern Regional Laboratory on the study of the utilization of a wider variety of agricultural products as ingredients in candy. This report contains details of new techniques for incorporating insoluble pulverized type ingredients into batches of candy, utilization of isolated peanut protein as an ingredient, and a report of the most recent "R" Series of experimental candies submitted to members of the Taste-Testing Committee.

No reprints available.

CONFECTIONERY FATS

2377. TEMPERING TRIGLYCERIDES BY MECHANICAL WORKING

Feuge, R. O.; Landmann, W.; Mitcham, D.; and Lovegren, N. V.  
J. Am. Oil Chemists' Soc. 39: 310-13. 1962.

The tempering of fat products to convert their components to stable polymorphs is an important and a sometimes troublesome problem in the manufacture of these products, particularly chocolate and chocolate-type confections. It has been found that a solid-to-solid transformation to the stable polymorphs can be effected by mechanical working consisting of extrusion under pressure. With a fat of relatively few components, such as cocoa butter,

evidence of the transformation can be obtained from X-ray diffraction patterns. For more complex fats, hardness and melting characteristics must be considered. There is evidence that mechanical working is also effective in the transformation of a cocoa butter-like fat made from hydrogenated cottonseed oil and olive oil, and in the transformation of highly hydrogenated cottonseed oil. Mechanical working to effect polymorphic transformation is also effective with products containing the fats mentioned.

2392. PROGRESS ON THE DEVELOPMENT OF CONFECTIONERY FATS. PART I.

TEMPERING OF CONFECTIONERY FATS AND COATING COMPOSITIONS  
(Published: conf. fat research shows...Mechanical Method  
May Give Tempered Effect)

Feuge, R. O.; Landmann, Werner; and Lovegren, N. V.  
Candy Ind. Technol. 119(2): 7-9. 1962. (No reprints available)

Fats in confectionery are tempered to develop the most desirable size and type of fat crystals so that the confectionery coating will be firm, glossy, of a fine surface texture, and resistant to fat bloom. Tempering usually consists of holding the coating for a short time at temperatures just below the melting point of the fat, followed by cooling. It has been observed that crystal modifications similar to those produced by tempering will occur on mechanical working of quickly-solidified crystals of fat. The mechanical working could be accomplished by forcing the fat repeatedly through small orifices at pressures up to 1,000 p.s.i. Evidence of the presence of the desired crystal structure was obtained by X-ray diffraction and hardness measurements. It is believed that through the use of this discovery improvements in the manufacture of chocolate and chocolate-type confections can be accomplished. Probably the use of tempering kettles can be eliminated, enrobing machines simplified, and a truly continuous process can be devised in which the material in actual process is perfectly tempered and always small in amount.

2393. PROGRESS ON THE DEVELOPMENT OF CONFECTIONERY FATS. PART II.

A NEW PROCEDURE FOR MAKING COCOA BUTTER-LIKE FATS  
(Published: cocoa butter-like fat...New Esterification  
Procedure Shows Promise)

Feuge, R. O.; Landmann, Werner; and Lovegren, N. V.  
Candy Ind. 119(3): 15-16. 1962. (No reprints available)

A new approach to the preparation of cocoa butter-like fats involves a procedure for esterification under conditions where all traces of moisture formed during the esterification reaction are removed as soon as they are formed and the random rearrangement of the fatty acids is prevented. 1,3-Distearin esterified with pure oleic acid yielded over 90% oleodistearin, of which about three-fourths was 1-oleodistearin.

Several cocoa butter-like fat products were prepared by esterifying oleic acid with the diglycerides of palmitic and stearic acids and fractionating the product by crystallization from hydrocarbon solvent. Physical properties agreed closely with that of cocoa butter. The newly discovered esterification procedure should be good for making cocoa butter-like fats from diglycerides which can be manufactured readily on a large scale. Additional work is underway to establish the best conditions for operating the process.

2442. DERIVATIVES OF FATS FOR USE AS FOODS

Feuge, R. O.

J. Am. Oil Chemists' Soc. 39: 521-27. 1962.

Suitable methods of manufacture always have been major factors in the development of new, edible products from fats. A review of the chemistry involved in the preparation of various surface-active agents, including monoglycerides, lactoglycerides, polyglycerol esters, and sugar esters, reveals a number of problems in controlling the course of the reaction to obtain economically products of reasonable purity and uniformity. Suggested innovations in the manufacture of some of these products are described. The production of specialty fats also presents some problems in chemistry. Because of such problems, cocoa butter-like fats have heretofore not been made from domestic oils. A recent discovery which may change this situation is discussed. The manufacture of specialty fats such as the acetoglycerides and brominated oils is described. A number of new derivatives of fats which have been made in the laboratory, and found to possess useful properties, are discussed from the standpoint of the chemistry involved in their preparation.

2300. COCOA BUTTER-LIKE FAT FROM COTTONSEED OIL: PRELIMINARY COST STUDY

Decossas, K.M.; Koltun, S.P.; Spadaro, J.J.; Feuge, R.O.; Pollard, E.F.; and Patton, E.L.

J. Am. Oil Chemists' Soc. 39: 146-50. 1962.

This paper includes a preliminary cost study for producing cocoa butter-like fat from hydrogenated cottonseed oil and triolein at an annual rate of 8.4 million lb. in a hypothetical plant, employing interesterification and fractional crystallization as used in pilot plant studies at the Southern Utilization Research and Development Division. Equipment costs, total plant cost, manufacturing costs, and general expenses are given. Operating cost is estimated at 36.9¢/lb. of product. This includes 31.8¢ for manufacturing, 4.1¢ for general expenses, and 1¢ for refining of the recycled mixture of saturates and unsaturates.

2176. COCOA BUTTER-LIKE SAMPLE. I. COATING WITH FAT NO. 5 HAS BETTER TEMPER.  
COCOA BUTTER-LIKE SAMPLE. II. FAT NO. 5 SHOWS LOW BLOOM TENDENCIES  
Feuge, R.O.; [Landmann, Werner; and Lovegren, N.V.]  
Candy Ind. & Confectioners J. 117(1): 34-35; (2):7-8. 1961.  
(No reprints available)

Progress being made in the development of a cocoa butter-like fat from domestic oils is described. An improved cocoa butter-like fat (No. 5) was prepared by modifying the procedure of interesterifying completely hydrogenated cottonseed oil with olive oil and separating the oleodisaturated glycerides by fractional crystallization. The separated fat melted mostly over the temperature range for cocoa butter, but contained less liquid phase below 68°F. and about 3.7% solids above 95°F., which is the approximate melting point of cocoa butter. Other physical characteristics were determined. It was concluded that a reasonably good cocoa butter-like fat can be prepared from domestic oils by an economically feasible process. While the cocoa butter-like fat was not an exact duplicate of cocoa butter and in some respects was less satisfactory, it was more desirable in some other respects. Among the more important was a relatively small variation in performance when conditions of melting and solidifying were changed.

2198. CONFECTIONERY FATS. I. PREPARATION BY INTERESTERIFICATION AND FRACTIONATION ON PILOT-PLANT SCALE  
Spadaro, J.J.; Lovegren, N.V.; Feuge, R.O.; and Patton, E.L.  
J. Am. Oil Chemists' Soc. 38: 461-65. 1961.

A cocoa butter-like fat has been prepared on a pilot-plant scale by the interesterification of hydrogenated cottonseed oil and a triolein product or olive oil followed by fractional crystallization from acetone at two different temperatures. The coproducts - a fraction which consists primarily of trisaturates and is obtained by fractionation at 20 to 28°C., and a fraction which is primarily di- and triunsaturates and is obtained from the low temperature (0°C.) filtrate - are reused in the process. In five of the six pilot-plant runs conducted, 100 pounds of 70:30 or 75:25 mixtures of the hard fat and liquid oil were used as starting materials. In the sixth run, 140 pounds were used. Yields varied from 25 to 35%. Characteristics of the cocoa butter-like fat products are discussed. Variations in the products were made by changing the ratio of starting materials to 75:25 and by lowering the first crystallization temperature from about 28° to about 19°C. Operational data obtained show that the process has commercial feasibility. Solvent-to-fat ratio was only 4 to 1. Filtration rates based on production of dry solids were 9 to 44 pounds per hour per square foot of filter area, respectively, for the first and second crystallizations. Although time to attain crystallization temperature was about 4 hours in the

pilot-plant operations, laboratory data indicate that comparable products can be obtained for crystallization times as low as one-half hour. The shorter crystallization time would be more applicable for commercial consideration. The steps in the process are considered conventional in commercial processing.

2199. CONFECTIONERY FATS. II. CHARACTERIZATION OF PRODUCTS PREPARED BY INTERESTERIFICATION AND FRACTIONATION  
Landmann, Werner; Lovegren, N.V.; and Feuge, R.O.  
J. Am. Oil Chemists' Soc. 38: 466-69. 1961.

Several cocoa butter-like fats, which had been prepared by fractional crystallization of the reaction product obtained on interesterifying highly-hydrogenated cottonseed oil and a triolein product or olive oil, were characterized and compared with cocoa butter. The fats, as obtained by fractional crystallization from acetone solutions, contained varying amounts of glycerides melting above 37°C., an undesirable feature which caused the fats to thicken too much when used in chocolate type compositions under the same conditions employed with cocoa butter. The higher-melting glycerides could be removed by filtration, or their proportions could be decreased by changing the fractionation temperatures. The fats melted mostly over the same temperature range associated with cocoa butter, and the best of the fats resembled cocoa butter closely over the temperature range 0° to 30°C. The cocoa butter-like fats resembled cocoa butter in hardness at all test temperatures. The fats were reasonably compatible with cocoa butter, that is, in mixtures of the two, one did not cause extensive premelting of the other. According to their cooling curves, the cocoa butter-like fats did not supercool as cocoa butter does. The former contain not only the 2-oleodisaturated glycerides of cocoa butter but also positional isomers of these glycerides. When the fats were molded under the same conditions employed with cocoa butter, linear shrinkage was only about one-third that of cocoa butter.

2077. METABOLIC STUDIES OF GLYCERIDE ESTERS OF ADIPIC ACID  
Shull, Rosemary L.; Gayle, Louis A.; Coleman, Richard D.;  
Alfin-Slater, Roslyn B. (University of Southern California);  
Gros, Audrey T.; and Feuge, R. O.  
J. Am. Oil Chemists' Soc. 38: 84-86. 1961.

Data on the digestibility, absorption, and in vivo oxidation of two types of adipic acid esters of glycerides, a diglyceride adipate and a polymer of fatty acids, adipic acid, and glycerol, have been presented. Findings indicate that both of these compounds have high digestibility coefficients and that the stearic acid moiety is well absorbed. However, although the stearic acid moieties are oxidized slowly in both cases, which is compatible with previous findings that a slow rate of in vivo oxidation of the stearic acid moiety of glycerides obtains, the rate of oxidation of the stearic acid is greater when fed as the diglyceride adipate than as the polyester.

2005. FIND MOLDING DIFFICULTY IN TEST COATINGS USING COCOA BUTTER-LIKE FAT *[Progress on Investigations of Confectionery Fats]*  
Feuge, R.O.  
Candy Ind. & Confectioners J. 115(4): 11-13,30. 1960.  
(No reprints available)

The article, which originally was submitted under the title "Progress on Investigations of Confectionery Fats", describes some of the research work on confectionery fats underway at the Southern Regional Research Laboratory. The preparation of several cocoa butter-like fats prepared from completely hydrogenated cottonseed oil and olive oil is discussed. The best of the cocoa butter-like fats possessed the short melting range, hardness at room temperature, and other desirable characteristics of cocoa butter. Unlike cocoa butter, none of the fats contracted to the desired degree on molding. Also, they possessed a relatively high viscosity when worked at the temperatures ordinarily used with cocoa butter. The preparation and characteristics of slab oils, which can be classed as another type of confectionery fat, also are discussed. The performance of an experimental slab oil consisting essentially of dibutyropalmitins and dibutyrostearins is described.

2037. PRODUCTION OF SPECIALTY EDIBLE FATS  
Feuge, R.O.  
J. Am. Oil Chemists' Soc. 37: 527-32. 1960.

This publication consists of a paper presented at the American Oil Chemists' Society Short Course on Edible Fats and Oils held at the University of Illinois in July 1960. The discussion is confined to several more or less arbitrarily selected products and processes. The requirements of cocoa butter-like fats are discussed in relationship to the properties and composition of cocoa butter. The suitability of cocoa butter-like fats from lauric acid oils and from oleic and linoleic acid oils is compared. The need for and preparation of oils having exceptional resistance to deterioration is discussed with emphasis on a laboratory preparation consisting of a mixture of dibutyropalmitins and dibutyrostearins. In the concluding section of the paper several potentially useful fats and oils are mentioned, including dibasic acid-containing fats, products from polyols other than glycerol, and acetoglycerides.

2046. MELTING AND DILATOMETRIC BEHAVIOR OF 2-OLEOPALMITOSTEARIN AND 2-OLEODISTEARIN  
Landmann, Werner; Feuge, R.O.; and Lovegren, N.V.  
J. Am. Oil Chemists' Soc. 37: 638-43. 1960.

The glycerides, 2-oleopalmitostearin and 2-oleodistearin, which are important components of confectionery fats, were synthesized, and their melting behavior and dilatometric properties were determined. Each glyceride was found to have four melting points: 18.2, 24.5, 33.0, and 37.4°C. for 2-oleopalmitostearin; 22.8, 30.0, 37.7, and 42.8°C. for

2-oleodistearin. The rate of transformation of the thermodynamically unstable polymorphs at temperatures just below their melting points was found to vary from a few seconds to more than eight days. For each glyceride, expansivities were determined for three polymorphs and melting dilations for two polymorphs. Mixtures of the two glycerides were examined dilatometrically and found to behave in some respects as single components. The mixtures were readily tempered so that both components were in the same polymorphic form, and expansivities and melting dilations were obtained for mixtures in three polymorphic forms. Dilatometric data also were obtained for a sample of cocoa butter and a sample of sweet milk chocolate of the coating type.

1908. PERMEABILITY OF SOME FAT PRODUCTS TO MOISTURE

Landmann, Werner; Lovegren, N.V.; and Feuge, R.O.  
J. Am. Oil Chemists' Soc. 37: 1-4. 1960.

Films of cocoa butter, highly hydrogenated cottonseed oil, mixtures of highly hydrogenated cottonseed oil and cottonseed oil, chocolate liquor, and sweet milk chocolate were prepared; and their permeability to water vapor was determined by the cup method. The permeability constant was calculated in terms of grams of water diffusing through a centimeter cube in one second under a vapor pressure gradient of one millimeter of mercury across the cube. Under the test conditions employed, the permeability constant for cocoa butter at room temperature was found to vary from  $5.8 \times 10^{-12}$  to  $81.6 \times 10^{-12}$ . The permeability constants for the highly hydrogenated cottonseed oil and the cocoa butter, under comparable conditions at room temperature, was found to be approximately  $1.3 \times 10^{-12}$  and  $33 \times 10^{-12}$ , respectively. From data obtained with cocoa butter it was concluded that the permeability constant increased with moderate increases in film thickness. Polymorphism was found to have a large effect on permeability, an approximately 15-fold difference was found between quickly chilled and tempered films of cocoa butter at 3 C. ( $37.4^{\circ}\text{F.}$ ). The percentage of liquid component in the fat was found to have a large effect on permeability. The increasing of the percentage of liquid cottonseed oil in highly hydrogenated cottonseed oil from 0 to 40% increased the permeability constant from  $1.3 \times 10^{-12}$  to about  $420 \times 10^{-12}$ . The permeability of chocolate liquor and sweet chocolate at room temperature was increased greatly when the relative humidity on the wet side of the films was increased to 100%. The non-fat components absorbed enough moisture to impair the structure of the film.

1865. EFFECT OF COMPOSITION AND POLYMORPHIC FORM ON THE HARDNESS OF FATS

Feuge, R.O.; and Guice, Wilma A.  
J. Am. Oil Chemists' Soc. 36: 531-34. 1959.

Hardness is an important index in the performance of confectionery and other fats. Using an instrument and technique which were essentially

a modification of those used in the Brinell test as applied to metals, the effect of composition and polymorphic form on the hardness of fats was investigated. It was found that the hardness of a given sample of fat was influenced by the degree of tempering to which the sample had been subjected. Hardness always increased as the components of a fat were converted to higher-melting polymorphs. However the hardest test specimens were not obtained with the highest tempering temperatures. Presumably the use of too high a temperature in tempering melted some of the lower-melting polymorphs and allowed them to resolidify in large crystals producing a softer matrix. Adding progressively larger amounts of one fat to another generally increased or decreased the hardness of the mixture in a more or less uniform manner. Adding small amounts of liquid oil to a hard fat greatly decreased the hardness index. Apparently the hardness index of a given fat decreases as the crystal size increases. It is believed that fats containing a sizable proportion of liquid component will become softer on prolonged storage because the presence of the liquid component makes possible a gradual increase in crystal size.

1870. NEW FAT PRODUCTS: GLYCERIDE ESTERS OF ADIPIC ACID  
 Ward, Truman L.; Gros, Audrey T.; and Feuge, R.O.  
 J. Am. Oil Chemists' Soc. 36: 667-71. 1959.

Six new glyceride esters of adipic acid were prepared, and their properties were determined. By the use of acetals and trityl groups the number, type, and positions of the fatty acid groups in the glycerol moiety were varied systematically. The reaction products were purified, their purities were established, and their melting points, viscosities, densities, and refractive indices were determined.

1872. POLYMERIC FATS FROM STEARIC, OLEIC, AND SHORT-CHAIN DIBASIC ACIDS  
 Feuge, R.O.; and Gros, Audrey T.  
 Ind. Eng. Chem. 51: 1019-22. 1959.

A need exists in the food industry for highly viscous oils and low-melting fats of exceptional resistance to oxidation. Such products can be prepared by polymerizing with the aid of short-chain dibasic acids the mono- and diglycerides derived from ordinary fats. Products prepared by the acylation of 1-mono-olein with one of the dibasic acids from the group fumaric, succinic, and adipic, possessed viscosities up to 325 centistokes at 50°C. and did not crystallize at temperatures below 0°C. Similar products prepared with stearic acid instead of oleic acid possessed viscosities up to 2360 centistokes at 50°C. The melting points ranged between 35.5 and 58.2°C. When the glycerol employed in the preparation was used in excess of that required to react with the carboxyl groups present, the viscosity and average molecular weight of the product decreased; and the melting point increased. The average molecular weight of the products never exceeded 1700. Theoretical considerations and experimental data indicated that no compounds having molecular weights above about three or four thousand were prepared. No experimental data are available on the physiological behavior of these compounds on ingestion.

1878. RECENT DEVELOPMENTS IN THE MODIFICATION OF FATS

Feuge, R.O.

Candy Ind. and Confectioners J. 113(2): 7, 12, 62; (3): 32,  
33, 36, 37. 1959.Published in two parts under titles: "Lab Studies Show  
Way to New Candy Fats" and "Lab Can Combine Selected  
Characteristics" (No reprints available)

The manuscript was submitted to the Candy Industry and Confectioners Journal under the title "Recent Developments in the Modification of Fats". The editor of the Journal divided the manuscript into two articles, one entitled "Lab Studies Show Way to New Candy Fats" and the other, "Lab Can Combine Selected Characteristics." The second article begins with the section of the manuscript entitled "Rearrangement and Directed Rearrangement." The first article describes in terminology understandable to non-chemists, the nature of fats, changes which can be made in their structure, and current practices in the fractionation of fats to produce products having more desirable characteristics. The second article describes other modification procedures; namely, rearrangement and directed rearrangement, esterification and hydrogenation. Probable future developments are forecast at the end of the second article.

## 1770. MOISTURE TRANSMISSION THROUGH FATS (Condensation)

Feuge, R.O.

Mfg. Confectioner 39(6): 79-80. 1959.  
(Reprints not available)

The permeability of a number of fat products to water vapor was measured. The cup method was employed. Permeability was calculated in terms of a permeability index, taking into consideration the surface area and thickness of the film of fat, the vapor pressure difference across the film, and the amount of water transferred per unit of time. The passage of moisture through films of fat under a given set of conditions was generally found to be relatively unaffected by moderate variations in the thickness of the film. The amount of moisture passing through a film under a given difference in vapor pressure sometimes was affected greatly by the relative humidities employed in the tests. Increasing the amount of liquid oil in a fat from 0% to 20% and above greatly increased the permeability index. The manner in which a fat was solidified and its subsequent heat treatment had a large effect on the permeability index. Seeding during solidification and tempering reduced the permeability to water vapor.

## 1763. ADDITION OF HYDROGENATED FATS TO CHOCOLATE TO IMPROVE HEAT RESISTANCE

Guice, Wilma A.; Lovegren, N.V.; Feuge, R.O.; and Coster, H.B.  
J. Am. Oil Chemists' Soc. 36: 4-8. 1959.

An investigation was made of the possibility of fortifying chocolate better to withstand summer temperatures by adding relatively small proportions of completely hydrogenated cottonseed oil and similar products.

Proportions of hydrogenated oils up to 10%, based on the weight of cocoa fat, do not affect mouthing quality significantly, yet they impart marked rigidity to the chocolate mass at temperatures at which cocoa fat softens and melts. Because the hard fat is practically insoluble in cocoa fat at the melting point of the latter, the hard fat, under the conditions adopted, does not alter the melting characteristics of the cocoa fat, and the short softening range of the cocoa fat is retained. The addition of the completely hydrogenated oil greatly retards the rate of fat leakage from chocolate at temperatures of about 36° C. (97° F.). The addition of small amounts of very hard fats to chocolate should be useful in improving the performance of molded bars. However if the modified chocolate is to be used for coating confections, the increase in viscosity which results on the addition of the hard fat presents a serious disadvantage.

1780. SEES COCOA BUTTER DUPLICATION

(Confectionery Fats of the Future)

Feuge, R.O.

Candy Ind. and Confectioners J. 112(12): 14-15, 131. 1959.

Changes which have occurred in the fat and oil industry during the past fifty years are described briefly. Using these changes as a yardstick and considering that the fat and oil industry is becoming more inclined to increase the complexity of processing operations in order to produce special, tailored products, future developments in the way of new fat products are suggested. The modification of domestic oil to duplicate cocoa butter, the most prized confectionery fat, is shown to be probable, as is the development of confectionery fats which are resistant to bloom and which will not soften at summer temperatures, yet will remain palatable. The development of superior, edible oils for use as slab dressings and as polishing oils in the manufacturing of confections is predicted, as well as the development of new and better food emulsifiers.

1774. PROGRESS ON INVESTIGATIONS OF CONFECTIONERY FATS, REPORT NO. 4

June 1, 1958 to June 1, 1959. (18 pp.) (Reprints not available  
Feuge, R. O.

Circular published and distributed by National Confectioners' Association, 36 Wabash Ave., Chicago 3, Ill.

A 140-pound sample of cocoa butter-like fat was obtained by combining the products obtained from 4 pilot-plant runs. The products from the 4 runs were practically identical, indicating that the process is reproducible. The composite Sample No. 1, had a slight flavor and odor which could have been removed if this had been deemed necessary. The sample also contained about 20% of trisaturated and other high-melting triglycerides, which it was believed might improve the performance of the fat at summer temperatures. Evaluation revealed that in candy formulations the fat was responsible for difficulties in tempering and demolding. Eating quality was below expectation. These faults were

believed to have been caused by the content of high-melting triglycerides. Subsequently, a portion of Sample No. 1 was modified by removing the 20% of fat melting above 98.6°F. This modified fat was better than Sample No. 1 but still not satisfactory. A second sample was prepared in the pilot plant. This second product, Sample No. 2, was sent to members of the Research and Development Committee for testing. The tests should be completed in the near future. Research on confectionery fats also was carried out in other areas. The investigation of the properties of the components of confectionery fats has revealed that 2-oleopalmitostearin, which comprises about 57% of cocoa butter, can solidify in four different forms; their melting points are 99.3, 91.4, 76.1, and 64.8°F. It also was found that 2-oleodistearin, which comprises about 22% of cocoa butter, solidifies in four forms, their melting points being 109.0, 99.9, 86.0, and 73.0°F. Expansivities and melting dilations of several of the forms were determined. One phase of the investigation of the transmission of moisture through confectionery fats has been completed. Additional research on the transmission of moisture through fats is now underway. The effect of composition and polymorphic form on the hardness of fats was investigated. It was found that crystal size and the amount of tempering which a fat receives greatly affects its hardness. The addition of a normally liquid oil to a hard fat greatly decreases the hardness. Several samples of a new type fat (essentially a mixture of dibutyrostearin and dibutylpalmitins) were prepared and submitted for testing as slab oils in the manufacture of candies.

1693. PROGRESS ON INVESTIGATIONS OF CONFECTIONERY FATS, REPORT NO. 3

November 1, 1957 to June 1, 1958. (16 pp.)

Feuge, R.O.

Circular published and distributed by National Confectioners' Association, 36 Wabash Ave., Chicago 3, Ill.

The NCA Fellow has determined the melting points of the two major components of cocoa fat. Each component was found to have three melting points. The amount of tempering required to shift from one melting point to the next varied from 80 seconds to more than 8 days. Melting dilations and volume changes accompanying changes from one solid form to another were measured. An investigation concerned with the passage of moisture through films of chocolate and confectionery fats was undertaken and is now almost completed. It was found that at low relative humidities milk chocolate and chocolate liquor are less permeable to moisture than is cocoa butter, while at high humidities the reverse is true. Passage of moisture through films of fat is affected greatly by the proportion of liquid oil in the fat. An instrument and technique have been developed for measuring precisely the hardness of a wide range of fats and waxes. The hardness of chocolate can also be measured. The degree of temper was found to have a large effect on the hardness of chocolate and cocoa butter. Experiments have shown that the addition of relatively small amounts of completely hydrogenated cottonseed oil will improve significantly the heat resistance of chocolate. Rigidity at elevated temperatures is increased while fat leakage is reduced. Mouthing quality and acceptability are hardly affected.

1688. 1,3-DIOLEIN AND 1,3-DISTEARIN ESTERS OF FUMARIC, SUCCINIC, AND ADIPIC ACIDS  
Feuge, R.O.; and Ward, T.L.  
J. Am. Chem. Soc. 80: 6338-41. 1958.

Certain of the dibasic acid esters of edible diglycerides should have properties making them desirable for a number of uses involving foods. To obtain information on such dibasic acid glycerides, the 1,3-diolein and 1,3-distearin esters of fumaric, succinic, and adipic acids were prepared by the reaction between the 1,3-diglycerides and the acid chlorides of the dibasic acids in the presence of pyridine or quinoline, special precautions being taken in most instances to reduce the amount of side reaction. The reaction products were purified and a number of their physical properties established. The melting points of the highest melting polymorphic forms of the distearin-containing compounds ranged from 82.1 to 89.9°C. The diolein-containing compounds either could not be obtained in a crystalline form or crystallization was incomplete, hence the melting points of these compounds were not determined. Through X-ray diffraction patterns it was established that the form obtained on quick chilling and the highest melting form of the distearin-containing compounds crystallized in a single-chain-length structure. The short spacings for the forms obtained on quick chilling were always equal to the short spacing characteristics of the alpha form of tristearin. The compounds crystallized as long needles, and in some instances clumps of crystals were obtained which resembled asbestos in appearance. All of the diglyceride esters of the dibasic acids were quite viscous when compared with cottonseed oil at the same temperature. The viscosity of the diolein-containing compounds ranged from 77.00 to 92.00 centistokes at 38.8°C. The viscosity of the distearin-containing compounds ranged from 11.42 to 18.93 centistokes at 98.8°C. On the basis of these data, diglyceride esters of the dibasic acids can be said to be approximately twice as viscous as cottonseed oil.

1683. AN INSTRUMENT FOR MEASURING THE HARDNESS OF FATS AND WAXES  
Lovegren, N.V.; Guice, W.A.; and Feuge, R.O.  
J. Am. Oil Chemists' Soc. 35: 327-31. 1958.

Heretofore a good method for measuring the hardness of fats and waxes has not been available. Instruments currently used to measure hardness (not consistency) are relatively inaccurate and give results based on an arbitrary scale. A new instrument and technique have been devised for measuring the hardness of fats and waxes. They are essentially an adaptation of the Brinell hardness test used for metals and alloys. In determining the hardness of a fat or wax, a perfectly round steel ball having a diameter as small as 0.1250 in. or as large as 0.5000 in. is pressed with a force of 200 g. to about 6 kg. onto the surface being tested. The hardness index, in terms of kilograms per square centimeter, is determined from the slight impression produced. The index is relatively independent of ball size, test load,

and other test conditions; provided these are confined to certain ranges. Hardness indices have been determined for products ranging from poorly tempered cocoa butter to rosin. Differences in hardness observed have varied over 1500-fold. The hardness of cocoa butter has been found to vary over seven-fold, depending on thermal history.

1623. THE PRESENT STATUS OF ACETOGLYCERIDES

Alfin-Slater, Roslyn B.; Coleman, R.D.; Feuge, R.O.; and Altschul, A.M.

J. Am. Oil Chemists' Soc. 35: 122-27. 1958.

Acetoglycerides can be prepared readily by several procedures, which are described. Fairly extensive investigations of the physiological behavior of acetoglycerides have been made. It is concluded, from work reported to date, that acetoglycerides have no deleterious effect on growth or on reproduction and that the mode of digestion of aceto-oleins and aceto-stearins does not differ appreciably from that of the corresponding natural fats. The acetyl group appears to be hydrolyzed and absorbed quickly and completely; no acetate has been demonstrated in the feces of acetoglyceride-fed rats. The rate of absorption of aceto-oleins exceeds that of acetostearins, but that for the latter is not unlike that of a commercial shortening. As to over-all digestibility, that of aceto-oleins, fed at levels up to 50% of the diet, appears to be equal to that of the corresponding normal triglycerides whereas the digestibility of acetostearins, when measured in animals pre-fed or simultaneously fed sufficient unsaturated fat to preclude essential fatty acid deficiency, is markedly better than the absorption of the normal saturated glycerides.

1614. COCOA BUTTER-LIKE FATS FROM DOMESTIC OILS

Feuge, R.O.; Lovegren, N. V.; and Cosler, H.B.

J. Am. Oil Chem. Soc. 35: 194-99. 1958.

Technical considerations indicate that cocoa butter-like mixtures can be prepared readily by the esterification of mixtures of oleic, palmitic, and stearic acids, or the interesterification of their glycerides, followed by the fractional crystallization of the reaction products. Using the indicated procedures, three cocoa butter-like fractions were prepared. One consisted essentially of oleopalmitostearins, another consisted essentially of oleodistearins, while the third consisted mostly of oleodipalmitins. On the basis of softening point curves, the oleopalmitostearin product was most compatible with cocoa butter, the oleodistearin product was the next most compatible, while the oleodipalmitin product was least compatible. When mixed with cocoa butter, all three of the products produced consistency vs. temperature curves whose shapes closely resembled that of cocoa butter. All of the mixtures softened over a short temperature interval though the actual temperature at which softening occurred varied. The several products are believed to be satisfactory cocoa butter replacements. Another cocoa butter-like fat was prepared by the interesterification of 70 parts of completely hydrogenated cottonseed oil and 30 parts of olive oil and the subsequent fractionation of the reaction product.

## 1535. NEW CONFECTIONERY FATS DATA READY: BOON TO INDUSTRY

Feuge, R.O.

Natl. Confectioners' Assoc., Publ. Conv. Rept. 26  
Rept. No. 2. 1957.

A description is given of the progress made by the National Confectioners' Association Fellow in preparing in a highly pure form and characterizing the two major components of cocoa butter and chocolate fat. The report also describes a new method being developed for measuring the hardness of confectionery fats and confections. Finally, mention is made of some successful attempts to fortify chocolate so that it is better able to withstand fluctuations in temperatures.

## 1530. FAT PRODUCTS RESEARCH. NEW, SPECIAL FATS FOR CONFECTIONERY USE

Feuge, R.O.

Candy Industry and Confectioners J. 109(10):21, 42-43, 53. 1957.

Several developments concerned with the preparation and characterization of edible fats, particularly confectionery fats, have been selected from published articles of the Southern Utilization Research and Development Division and are described. From data obtained in the characterization of acetoglycerides, it is shown how dilatometry, a field in which the Laboratory has contributed significantly, provides a number of items of important information, including coefficient of expansion, melting dilations, polymorphic transformations and volume changes accompanying polymorphic transformations. A new instrument and technique developed for measuring the hardness of fats and waxes is described and discussed. Among new products developed at the Laboratory, the following are described: Cocoa butter-like fat made from domestic oils, acetoglyceride products and their unique properties, and new-type polymeric fats made from domestic fats and short-chain dibasic acids.

## 1525. THE COMPOSITION OF BLOOM FAT IN CHOCOLATE

Cerbulis, J.; Clay, C.; and Mack, C.H.

J. Am. Oil Chem. Soc. 34: 533-37. 1957.

The composition of the fat which has bloomed in chocolate has been investigated. There is evidence that the type of center has no effect upon the fatty-acid composition of the bloom fat. The rate of blooming, however, was found to be affected by the center. The theory that the oils in coated nuts migrate to the chocolate surface to cause fat bloom has been disproved. Comparison of the fatty acids present in the bloom fat with respect to the coating fat show a decrease in the unsaturated acids and an increase in the saturated acids. There is evidence that hydroxyl-containing oxidation products of the fatty materials in the chocolate are not present in the migrated fat.

1473. PHYSICAL PROPERTIES OF ACETO- AND BUTYRO-OLEINS, MONO-OLEIN, AND DIOLEIN  
 Gros, A.T.; and Feuge, R.O.  
 J. Am. Oil Chem. Soc. 34: 239-44. 1957.

Certain di- and triglycerides containing both long and short chain fatty acids are liquid at low temperatures and exhibit unique properties. Four of these compounds; 1-aceto-3-olein, 1,2-diaceto-3-olein, 1-butyro-3-olein and 1,2-dibutyro-3-olein, were prepared and some of their physical properties were determined. For purposes of comparison, data on 1-mono-olein and 1,3-diolein were obtained. Melting points of the highest melting forms ranged from -29°C. for the 1,2-dibutyro-3-olein to 32°C. for the 1-mono-olein. Melting points for the lower-melting polymorphic forms of some compounds were not established definitely because of limitations of existing equipment. Expansibilities in the liquid state ranged from 0.000812 ml./g./°C. for the 1-mono-olein to 0.000912 ml./g./°C. for the 1,2-diaceto-3-olein. Specific volumes in the liquid state at 35°C. ranged from 1.0392 ml./g. for the 1,2-diaceto-3-olein to 1.0955 ml./g. for the 1,3-diolein.

1472. PROGRESS ON INVESTIGATIONS OF CONFECTIONERY FATS, REPORT NO. 1, August 1, 1956 to April 30, 1957. (10 pp.)  
 Landmann, Werner  
 Circular published and distributed by National Confectioners' Association of the United States, Inc., 221 N. LaSalle St., Chicago 1, Ill.

Several highly pure samples of 2-oleodistearin and 2-palmito-oleostearin, two fat compounds which constitute about 80% of cocoa butter, have been prepared, and their preparation is described. Characterization of these pure fats is being undertaken. An apparatus has been designed and fabricated, and a technique is being developed, which will enable the accurate measurement of the hardness and softening range of confectionery fats and confections. In still another phase of the investigation, experiments have demonstrated that addition of a small percentage of completely hydrogenated cottonseed oil markedly improves the ability of chocolate to withstand temperatures of 100-105°F. with little effect on the mouthfeel quality.

1392. PROGRESS IN A NEW AREA OF CANDY RESEARCH  
 Altschul, A.M.  
 Natl. Confectioners Assoc., 73rd Ann. Convention Report, 19-21. 1956.

Research on the chemistry of fats and oils as a means of improving their properties for use in the making of candies is discussed. Knowledge of the general chemistry of fats and oils has already sufficiently advanced to justify belief that through study of the relationship between changes in composition and physical properties of the fat, cocoa

butter can be reproduced, or a similar fat produced which has specific properties more suitable for certain types of candies. For example, fats may be produced compatible with cocoa butter, but with higher melting points for use in summer candies. Other physical properties, such as moisture transfer, compatibility, melting point, and polymorphic form, may also be improved through greater knowledge of the chemistry of fats.

1391. PERMEABILITY OF ACETOSTEARIN PRODUCTS TO CARBON DIOXIDE, OXYGEN, AND NITROGEN  
 Lovegren, N.V.; and Feuge, R.O.  
 Agr. & Food Chem. 4: 634-38. 1956.

The acetostearins are modified fats capable of being formed into waxlike films, potentially useful as protective coatings. Permeability to carbon dioxide was measured by modification of the standard cup method, using Ascarite as the absorbent. An apparatus was developed which could measure in about 8 hours the passage of a few hundredths of 1 ml. of gas through a film of acetostearin, 2-3/8 inches in diameter, into a sweep gas which was analyzed. Permeability of the acetostearins to carbon dioxide was less than that reported by other investigators for ethylcellulose and approximately the same as for polystyrene and polyethylene. Nylon and regenerated cellulose have lower permeabilities. Polystyrene and ethyl cellulose are more permeable to oxygen and nitrogen than acetostearin; polyethylene has approximately the same permeability. Cellulose acetate, regenerated cellulose, and nylon are less permeable.

1389. DILATOMETRIC PROPERTIES OF SOME BUTYROPALMITINS AND STEARINS AND ACETOPALMITINS  
 Feuge, R.O.; and Lovegren, N.V.  
 J. Am. Oil Chem. Soc. 33: 367-71. 1956.

Certain di- and triglycerides containing both short and long chain fatty acids normally solidify to waxy solids and as such have a number of potential uses. Six of these compounds, namely; 1-butyro-3-palmitin; 1,2-dibutyro-3-palmitin; 1-butyro-3-stearin; 1,2-dibutyro-3-stearin; 1-aceto-3-palmitin; and 1,2-diaceto-3-palmitin have been prepared and some of their physical properties have been determined. Expansibilities in the solid and liquid states, melting dilations, densities, melting points, points of transition from one polymorphic form to another, and ease of tempering to the higher polymorphic forms were measured or calculated. Melting dilations for the highest melting form of each compound ranged from 0.612 ml. to 1.203 ml. per 10 grams. The 1,2-dibutyro-3-palmitin changed very slowly to its high melting form (m.p., 10 C.). On the other hand the 1-butyro-3-palmitin and 1-butyro-3-stearin quickly temper to the high melting forms.

GENERAL

1934. A DECADE OF SUGARCANE PROCESSING RESEARCH IN LOUISIANA  
 Martin, L.F.; Guilbeau, W.F.; Fort, C.A.; Roberts, E.J.;  
 Smith, B.A.; Coll, E.E.; Jackson, J.T.; Friloux, J.J.; and  
 Cashen, N.A.  
 Sugar J. 22(11): 11, 13-14, 16, 19-20. 1960.

The article reviews briefly work of the Sugarcane Investigations during the past ten years, on pilot-plant scale testing of new varieties of cane to ensure their suitability for processing; on composition of sugarcane juice and methods of analysis used to obtain accurate quantitative determinations; on pilot-plant experiments with radioactive tracer labeled glucose to study the extent to which glucose decomposes or reacts during processing; and on the use of ion-exchange in the purification of sugarcane juice and sirup. Four tables.

1480. RESEARCH ON SUGARCANE PROCESSING AND UTILIZATION  
 Martin, L.F.  
 Sugar J. 19(11): 22-23. 1957.

This article reports briefly on the progress of work underway in the Sugarcane Products Section of the Southern Utilization Research and Development Division of ARS, for the past two seasons. Topics discussed are determination of the processing quality of the new sugarcane varieties and basic studies of the dependence of processing quality upon composition; search for means of obtaining more sugar per ton of cane and investigating methods of producing sugar of direct consumption quality; and improving confectionery products to expand the use of sugar.

1249. SOUTHERN UTILIZATION RESEARCH BRANCH SOLVES PROBLEMS OF SUGARCANE INDUSTRY  
 Paringer, K.L.; and Guilbeau, W.F.  
 Ind. Lab. 6(12): 6-9. 1955.

Work of the Southern Utilization Research Branch on sugarcane and its products is described. The Processing Unit studies engineering problems in the processing of sugarcane and the manufacture of by-products, and investigates the processing and evaluation of new varieties. A field laboratory at Houma, La., works on clarification problems, and filterability of sugars, and carries out chemical analyses for constituents of sugarcane products. The Products Unit conducts fundamental and analytical research, comprising studies of composition of the sugarcane plant, juices, and their products, and investigates recovery of byproducts from cane juices and molasses and their uses, and seeks improved utilization of sugar and other agricultural products in candy manufacture. Among developments mentioned are ion-exchange sugar, and a practical method for separation of aconitic acid.

## 608. THE PRODUCTION AND USE OF SUGARCANE

Martin, L.F.

U. S. Dept. Agr., Yearbk. of Agr., 293-99.

1950-1951.

Sugarcane, the source of almost three-fourths of the world's supply of sugar and the source of many other products, intrigues chemists because sugar is the cheapest and most abundant pure organic chemical available to industry. Following a discussion of production areas, some of the problems facing the cane-sugar industry are listed. Processes of producing sugar from cane are described and developments in producing new varieties of cane and the changes in milling practices needed to process the new varieties are discussed. The role of chemical research in helping the industry meet the problems introduced by the adoption of mechanical harvesting is evaluated. The value of bagasse fiber and sugarcane wax, byproducts of sugar production, and the potentialities for discovering new products are pointed out, especially the promise for commercial uses shown by the new byproduct, aconitic acid. Present industrial uses of sugar are listed.

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